RMZ

MATERIALS and GEOENVIRONMENT

MATERIALI in GEOOKOLJE

RMZ – M&G, **Vol. 60** No. 4
pp. 231–302 (2013)

Ljubljana, December 2013

RMZ – Materials and Geoenvironment
RMZ – Materiali in geookolje
ISSN 1408-7073

Old title/Star naslov
Mining and Metallurgy Quarterly/Rudarsko-metalurški zbornik
ISSN 0035-9645, 1952–1997

Published by/Izdajatelj
Faculty of Natural Sciences and Engineering, University of Ljubljana/
Naravoslovnotehniška fakulteta, Univerza v Ljubljani

Associated Publisher/Soizdajatelj
Institute for Mining, Geotechnology and Environment, Ljubljana/
Inštitut za rudarstvo, geotehnologijo in okolje
Velenje Coal Mine/Premogovnik Velenje

Editorial Address/Naslov uredništva
RMZ – Materials and Geoenvironment
Aškerčeva cesta 12, p. p. 312
1001 Ljubljana, Slovenija
Tel.: +386 (0)1 470 45 00
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Design and DTP/Oblikovanje, prelom in priprava za tisk
IDEJA za ITGTO

Print/Tisk
Birografika BORI, d. o. o.
Printed in 300 copies./Naklada 300 izvodov.

Published/Izhajanje
4 issues per year/4 številke letno

Partly funded by Ministry of Education, Science and Sport of Republic of Slovenia./Pri financiranju revije sodeluje Ministrstvo za izobraževanje, znanost in šport Republike Slovenije.

Articles published in Journal "RMZ M&G" are indexed in international secondary periodicals and databases:/Članki, objavljeni v periodični publikaciji „RMZ M&G", so indeksirani v mednarodnih sekundarnih virih: Civil Engineering Abstracts, CA SEARCH® – Chemical Abstracts® (1967–present), Materials Business File, Inside Conferences, ANTE: Abstract in New Technologies and Engineering, METADEX®, GeoRef, CSA Aerospace & High Technology Database, Aluminium Industry Abstracts, Computer and Information Systems, Mechanical & Transportation Engineering Abstracts, Corrosion Abstracts, Earthquake Engineering Abstracts, Solid State and Superconductivity Abstracts, Electronics and Communications Abstracts.

Annual subscription for individuals in Slovenia: 16.69 EUR, for institutions: 22.38 EUR. Annual subscription for the rest of the world: 20 EUR, for institutions: 40 EUR/Letna naročnina za posameznike v Sloveniji: 16,69 EUR, za inštitucije: 33,38 EUR. Letna naročnina za tujino: 20 EUR, inštitucije: 40 EUR.

Current account/Tekoči račun
Nova ljubljanska banka, d. d. Ljubljana: UJP 01100-6030708186

VAT identification number/Davčna številka
24405388

Online Journal/Elektronska revija
www.rmz-mg.com

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Kazalo

Historical Review

More than 90 years have passed since the University Ljubljana in Slovenia was founded in 1919. Technical fields were united in the School of Engineering that included the Geologic and Mining Division, while the Metallurgy Division was established only in 1939. Today, the Departments of Geology, Mining and Geotechnology, Materials and Metallurgy are all part of the Faculty of Natural Sciences and Engineering, University of Ljubljana.

Before World War II, the members of the Mining Section together with the Association of Yugoslav Mining and Metallurgy Engineers began to publish the summaries of their research and studies in their technical periodical Rudarski zbornik (Mining Proceedings). Three volumes of Rudarski zbornik (1937, 1938 and 1939) were published. The War interrupted the publication and it was not until 1952 that the first issue of the new journal Rudarsko-metalurški zbornik – RMZ (Mining and Metallurgy Quarterly) was published by the Division of Mining and Metallurgy, University of Ljubljana. Today, the journal is regularly published quarterly. RMZ – M&G is co-issued and co-financed by the Faculty of Natural Sciences and Engineering Ljubljana, the Institute for Mining, Geotechnology and Environment Ljubljana, and the Velenje Coal Mine. In addition, it is partly funded by the Ministry of Education, Science and Sport of Slovenia.

During the meeting of the Advisory and the Editorial Board on May 22, 1998, Rudarsko-metalurški zbornik was renamed into "RMZ – Materials and Geoenvironment (RMZ – Materiali in Geookolje)" or shortly RMZ – M&G. RMZ – M&G is managed by an advisory and international editorial board and is exchanged with other world-known periodicals. All the papers submitted to the RMZ – M&G undergoes the course of the peer-review process.

RMZ – M&G is the only scientific and professional periodical in Slovenia which has been published in the same form for 60 years. It incorporates the scientific and professional topics on geology, mining, geotechnology, materials and metallurgy. In the year 2013, the Editorial Board decided to modernize the journal's format.

A wide range of topics on geosciences are welcome to be published in the RMZ – Materials and Geoenvironment. Research results in geology, hydrogeology, mining, geotechnology, materials, metallurgy, natural and anthropogenic pollution of environment, biogeochemistry are the proposed fields of work which the journal will handle.

Editor-in-Chief

Zgodovinski pregled

Že več kot 90 let je minilo od ustanovitve Univerze v Ljubljani leta 1919. Tehnične stroke so se združile v tehniški visoki šoli, ki sta jo sestavljala oddelka za geologijo in rudarstvo, medtem ko je bil oddelek za metalurgijo ustanovljen leta 1939. Danes oddelki za geologijo, rudarstvo in geotehnologijo ter materiale in metalurgijo delujejo v sklopu Naravoslovnotehniške fakultete Univerze v Ljubljani.

Pred 2. svetovno vojno so člani rudarske sekcije skupaj z Združenjem jugoslovanskih inženirjev rudarstva in metalurgije začeli izdajanje povzetkov njihovega raziskovalnega dela v Rudarskem zborniku. Izšli so trije letniki zbornika (1937, 1938 in 1939). Vojna je prekinila izdajanje zbornika vse do leta 1952, ko je izšel prvi letnik nove revije Rudarsko-metalurški zbornik – RMZ v izdaji odsekov za rudarstvo in metalurgijo Univerze v Ljubljani. Danes revija izhaja štirikrat letno. RMZ – M&G izdajajo in financirajo Naravoslovnotehniška fakulteta v Ljubljani, Inštitut za rudarstvo, geotehnologijo in okolje ter Premogovnik Velenje. Prav tako izdajo revije financira Ministrstvo za izobraževanje, znanost in šport.

Na seji izdajateljskega sveta in uredniškega odbora je bilo 22. maja 1998 sklenjeno, da se Rudarsko-metalurški zbornik preimenuje v RMZ – Materiali in geookolje (RMZ – Materials and Geoenvironment) ali skrajšano RMZ – M&G. Revijo RMZ – M&G upravljata izdajateljski svet in mednarodni uredniški odbor. Revija je vključena v mednarodno izmenjavo svetovno znanih publikacij. Vsi članki so podvrženi recenzijskemu postopku.

RMZ – M&G je edina strokovno-znanstvena revija v Sloveniji, ki izhaja v nespremenjeni obliki že 60 let. Združuje področja geologije, rudarstva, geotehnologije, materialov in metalurgije. Uredniški odbor je leta 2013 sklenil, da posodobi obliko revije.

Za objavo v reviji RMZ – Materiali in geookolje so dobrodošli tudi prispevki s širokega področja geoznanosti, kot so: geologija, hidrologija, rudarstvo, geotehnologija, materiali, metalurgija, onesnaževanje okolja in biokemija.

Glavni urednik

Original scientific paper

Received: October 19, 2013
Accepted: December 1, 2013

Influence of process parameters on hydrogen content in steel melt

Vpliv procesnih parametrov na vsebnost vodika v jekleni talini

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Abstract

Hydrogen content in steel melt plays an important role in determination of mechanical properties of solidified steel. In the case of too high amount of hydrogen annealing has to be applied. This process is time and money consuming.

During steel production the majority of hydrogen is removed from steel melt during the vacuum degasing process. The amount of hydrogen in steel melt after vacuuming is not only dependent on time of degasing but also other technological parameters.

Neuronal networks were used for analyses of technological parameters with influence on the hydrogen content in melt. Also their importance was evaluated. Further step towards prognostication of hydrogen content in steel melt was done with comparison between the results of predictions calculated on the basis of different databases.

As the most important factor was recognized the absolute humidity of air (AH) what is also documented in literature[1]. It was proven that the prediction accuracy has not drastically got worse if air temperature was used instead AH. Other technological parameters have minor, but not negligible influence on hydrogen content in steel melt.

Key words: hydrogen content, steel melt and neuronal network

Izvleček

Vsebnost vodika v jekleni talini močno vpliva na mehanske lastnosti jekla v trdnem stanju. Pri preveliki vsebnosti je potrebno žarjenje, kar pomeni daljši čas in večje stroške izdelave.

Večino vodika se iz jeklene taline odstrani med vakuumskim razplinjenjem. Vsebnost vodika po razplinjenju ni odvisna samo od časa razplinjenja, ampak tudi drugih parametrov.

Vpliv tehnoloških parametrov smo analizirali in ovrednotili z uporabo nevronskih mrež. Nadaljnji korak pri napovedovanju vsebnosti vodika v talini je bil narejen s primerjavo izračunov, narejenih na osnovi različnih podatkovnih baz.

Najpomembnejši vplivni faktor je absolutna vlaga zraka, kar je v skladu s podatki v literaturi[1]. Ugotovili smo, da zamenjava najpomembnejšega parametra z drugim, temperaturo zraka, drastično ne poslabša napovedi. Drugi tehnološki parametri imajo manjši, ampak ne zanemarljiv vpliv na vsebnost vodika v jekleni talini.

Ključne besede: vsebnost vodika, jeklena talina in nevronske mreže

Introduction

Hydrogen in steel can cause a lot of defects what is very well documented in the literature. Over 40 articles can be finding in Science Direct with key words "steel", "hydrogen" and "defect" solely for the year 2013. Some defects caused by hydrogen content in steel[2, 3] can be repaired with thermal treatment, which is particularly in the case of large ingots very time consuming and also uneconomic. Better way to deal with the problem of too high hydrogen content in steel melt is to reduce it during the process of secondary steel making and with appropriate casting technology.

To control the amount of hydrogen during steelmaking it is necessary to identify the parameters, which have the main influence. Several studies were conducted worldwide in last few years, which give some insight in the problematic, i.e. from S. Misra[1], H. E. Hurst[4] and R. J. Fruehan[5].

Also in Slovenian steel-works the risk of too high hydrogen content in steel melt exists. The most seriously is this problem indicated at casting of large ingots. This problematic was presented in works of the researchers from Metal Ravne.[6, 7]

In the past few years' studies with the intention to identify the influential parameters and consequently to lower the hydrogen amount in the steel melt were made also in collaboration between Department of Materials and Metallurgy and Slovenian steel-works.[8–10]

Mentioned studies have identified only the influence of separate parameters. Our experiences with neuronal networks, which were successfully employed in case of characterisation and prediction of technological parameters[11–13], have encourage us to find the correlation between them.

Experimental work

For the analyses and predictions program Statistica Neuronal Networks was used which enables use of various types neuronal networks and this makes it suitable for solving problems from different areas, for regression as well as classification cases. In the case of hydrogen content prediction – regression problem, MLP (multilayer perceptrons) type neuronal network was used. The schematic representation of this neuronal network with three layers is shown at Figure 1.

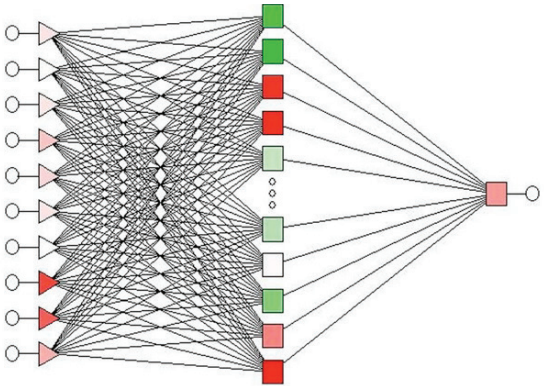

Figure 1: *MLP neuronal network with ten input parameters in first layer, fifteen neurons in hidden layer and one output parameter.*[11]

For usage of neuronal nets the reliable database has to be collected and prepared for further work. Some measurements from the working process are automatically saved into the centralised database therefore the majority of necessary data was already in the system.

In the case of industrial measurements obtaining the representative database is always the important step. If data base is incomplete, unreliable or even wrong, parameters, which do not have any correlation with hydrogen content in steel melt could be regarded as crucial. Measurements and construction of initial data base was carried-out at Metal Ravne.

From the bulk database, which was composed from automatically included measured data as well as manually inserted records the uniform table was made. This table was built-up from 14 parameters (2 outputs and 12 inputs) and more than 2 500 records, i.e. data vectors.

The hydrogen content in steel melt after vacuum treatment was measured with two methods, with direct measurements with Hydris device[4] and indirect with chemical analysis from solid sample.[8] Hydris measurements were estimated as more reliable and thus used in this study. This decision has decreased number parameters to 13 and number of records to 1 771.

Another step was filtration of the database. The filtration process was made with the help of basic statistic tools that has enabled identification

of data with unreliable extreme values. In spite of the fact that neuronal networks can very good compensate some inconstancies in data the false data on the boundary of the analysed area can cause unrealistic predictions. This filtering process also did not have big influence on the number of available data, only a little more than 1 % of records were excluded.

From the remaining data table with 1 749 and 13 parameters were built. As target parameter – output data, hydrogen content measured with Hydris device was selected.

It is clear that steel grade could have some influence on the hydrogen content in the steel melt but it can be indirectly described with other parameters from data base.

After these reductions final database was formed. It consists of 1 749 records, so-called data vectors, with 12 parameters (11 inputs and 1 output). Database was then randomly divided into training, test and verification databases in ratio 75 : 15 : 10 what is commonly practice in analysis with neuronal networks.

First neuronal networks were used on the whole database and analysis of impact of parameters was made (Table 1). From this table it can be clearly seen that most influential parameter is absolute humidity of air (AH) what is in accordance with the results described in work of Misra.[1]

Because of very scattered data in training database what can be clearly seen at Figure 2 the very accurate predictions are not possible, i.e. generalisation is expected. With this in mind the predictions of hydrogen content in steel melt were first made only with one input parameter – absolute humidity of air. Regarding to Table 1 also temperature (T), time of vacuum treatment (t_{vacuum}) and mass of lime (m_{CaO}) have certain influence on hydrogen content in steel melt and thus better predictions can be expected if they were included. These parameters were in fact taken into account in different combinations. For the final estimation of the maximum accuracy of the hydrogen content description all 11 input parameters were used in neuronal network analysis.

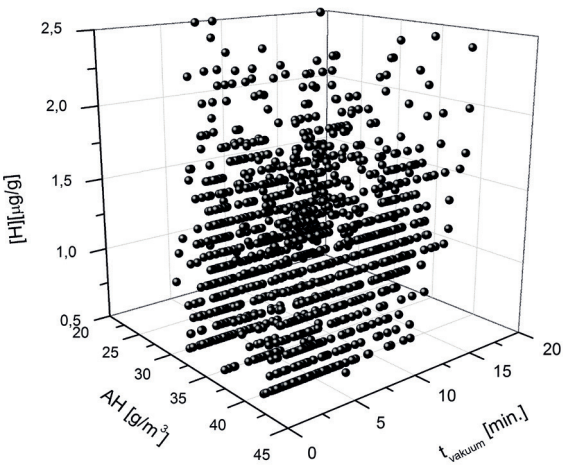

Figure 2: *Influence of absolute humidity of air and time of degassing on the hydrogen content in steel melt.*

Results and discussion

The successfulness of prediction is commonly defined with the least squares method (R^2) and this measure was also applied in this study. Comparison between predictions with different neuronal nets trained with various numbers of input parameters is presented in following subsections.

Influence of restricted database on the hydrogen content

When industrial data is used as input parameter for predictions it is possible that some of the regularly used parameters are not available. To find out if replacements of normally used input parameters are possible and how they affect the accuracy of predictions trials with different number of input parameters were made.

Table 1: *Sensitivity analysis of influential parameters*

m (kg)	$t_{process}$ (min)	t_{vacuum} (min)	T (°C)	m_{CaC_2} (kg)	m_{boksit} (kg)	m_{Al} (kg)	m_{ferro} (kg)	m_{carbur} (kg)	m_{CaO} (kg)	AH (g/m³)
1.004 2	1.005 3	1.042 5	1.067 0	1.001 9	1.004 3	1.012 4	1.000 8	1.016 8	1.050 2	1.136 8

— *Absolute humidity of air*
In this case only absolute humidity of air was used as input parameter. From the results presented in Table 1 is clear that this parameter has the most important role in the prediction of hydrogen content. Despite quite big scattering of measured data the R^2 for these predictions was 0.433.

— *Absolute humidity of air and air temperature*
From the results of neuronal network training collected in Table 1 the second most influential parameter is air temperature. Air temperature and absolute humidity of air are not entirely independent what can be seen from Figure 3 and that is why the correlation between measured and predicted values has not drastically changed; R^2 for these predictions was 0.440.

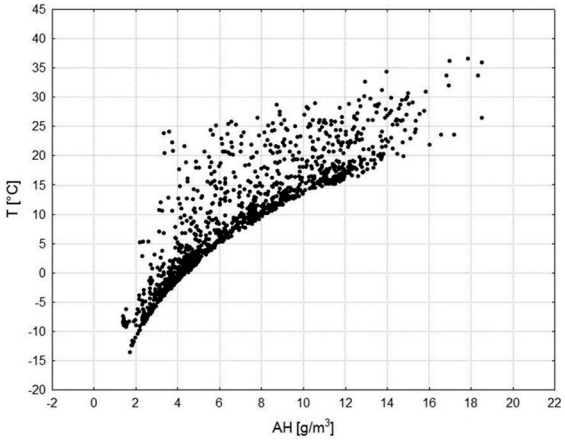

Figure 3: *Relationship between absolute humidity of air and temperature.*

— *Absolute humidity of air and lime mass*
With only two input parameters, absolute humidity of air and mass of added lime, the correlation between the measured and with neuronal network predicted data was significant better than with only one parameter – absolute humidity of air. The correlation factor was 0.488.

— *Absolute humidity of air, lime mass and time under low pressure (vacuum)*
From the logical point of view it is clear that the time in which the melt is under low pressure must have an influence on the hydrogen content. The results of the predictions with neuronal nets have confirmed that with improved accuracy and correlation factor rises to value 0.513.

— *Predictions without absolute humidity of air as input parameter*
Two different predictions were made. In first predictions temperature of air and mass of lime were used as input parameters. The correlation factor was 0.451 what is slightly better than result when only absolute humidity of air was used as input parameter and at the same time less accurate as combination of absolute humidity of air and lime mass.
For second predictions mass of lime and time of steel melt under low pressure were used as input. The correlation factor 0.222 suggests that those two parameters don't have major influence on the hydrogen content in the steel melt.

Influence of all eleven input parameters on the hydrogen content

It is quite common that for the prediction all available data is used. The correlation factor was 0.553 what is understandingly better than at all previously mentioned efforts. Because of the scattered input data and thus quite big generalisation of predictions the risk of overtraining is not present. This can be also deduced from comparison of correlation factors for train, test and verification database. The results with weighted averaged correlation factor are presented in Table 2.

Table 2: *The least squares values for three databases with 11 input parameters*

Train	Test	Verification	Average
0.555	0.534	0.515	0.548

Hydrogen content as function of various parameters

The influence of absolute humidity of air was found as predominant. In this analysis further three parameters were studied: temperature of the air, the mass of lime added for slag forma-

tion and time of low pressure. The influence of these three parameters together with the main parameter are presented and discussed in further paragraphs.

— *Influence of air temperature*

The hydrogen content in steel melt is bigger in the case of higher absolute humidity of air. The lowest limit of absolute humidity of air increases with the air temperature growth as can be seen from Figure 3. That is why increase of hydrogen content is in steel melt with rising air temperature expected and logical. The graphical interpretation is presented at Figure 4.

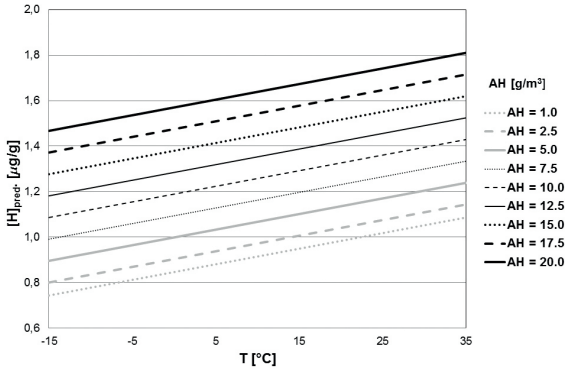

Figure 4: *The influence of air temperature and absolute humidity of air on the hydrogen content.*

— *Influence of added mass of lime*

Lime usually contains some amount of moisture. At elevated temperatures and in the presence of carbon hydrogen can be produced from steam[14], consequently the amount of hydrogen in melt increases. The rise in the hydrogen content in the steel melt with the larger amount of added lime is clearly noticeable at the Figure 5.

From the diagram it can be deducted that the gradient of hydrogen content increase is bigger at smaller values of added lime mass. Also the influence of absolute humidity of air is visible. In the case of lower amounts of absolute humidity of air the increase of hydrogen content is evident at whole region. On the other hand at high values of absolute humidity of air increase of hydrogen content in melt is restricted only on the difference between addition and no addition.

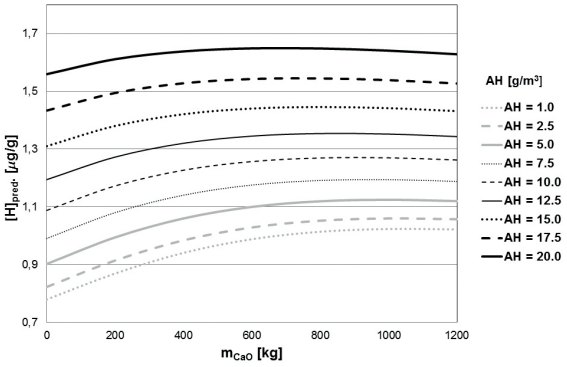

Figure 5: *The influence of added mass of lime and absolute humidity of air on the hydrogen content.*

— *Influence of time under low pressure*

The longer times of vacuuming logically leads to the lower values of hydrogen content in the melt. At the diagram at Figure 6 this assumption is confirmed.

The analysis also shows one unexpected result. It was presumed that with longer times the change in the hydrogen content will become slower but the results does not confirm that assumption.

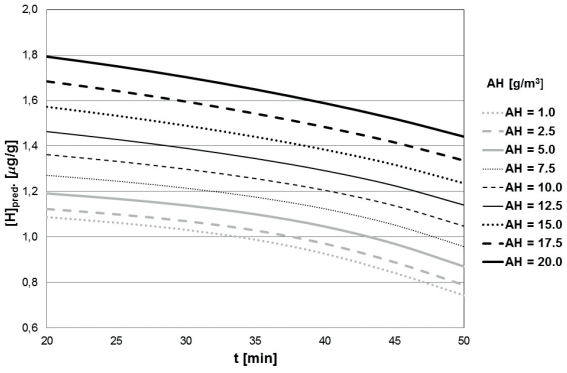

Figure 6: *The influence of time of degassing and absolute humidity of air on the hydrogen content.*

Comparison with the references

The hydrogen content in our study was measured after the degasing process unlike the most of data published in literature. In spite of that the conclusions from this study are in agreement with the literature. The influence of absolute humidity of air is predominant in this study and also in study from Misra.[1] Also the rise of hydrogen content in the steel mold with increased amount of lime is in agreement with results published by Fruehan.[5]

Conclusions

The very good correlation between the measured hydrogen content in steel melt and results of neuronal networks prediction was not expected. The main reason is very big scatter in measured results. The predictions were thus more global and orientated into the estimation of loose rules which can help to predict the tendencies of hydrogen amount change during the process of steelmaking.

Some conclusions from the analysis:

- Absolute humidity of air is the parameter which has the most important influence on the hydrogen content in the steel melt. The drastic change of other influential parameters can compensate only smaller changes in absolute humidity of air.
- Temperature is in correlation with the absolute humidity of air and thus can be alternatively used as input parameter in the case of lack of data for absolute humidity of air.
- Despite the fact that other measured parameters, e.g. mass of added lime, time under low pressure, have minor influence the prediction accuracy increases. On the other hand with only this data as input parameter the prediction of hydrogen content are not possible.
- At higher air temperatures higher values of hydrogen content in the melt are expected.
- Addition of lime during steelmaking also increases the amount of hydrogen in the melt. The rise is more noticeable at lower masses and lower absolute humidity of air.
- Longer times of vacuuming (melt under low pressure) leading to the lowering hydrogen content in the steel melt.

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Review paper

Received: October 10, 2013
Accepted: November 28, 2013

Interfaces in the magnesium-matrix composites

Mejna območja v kompozitih z magnezijevo osnovo

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Abstract

Processes at the matrix/reinforcement interfaces strongly influence the properties of the composites. The basic task of the interfaces is to assure the strong bonding between the composite's constituents. In addition, they must be mechanically and thermodynamically stable. Therefore, the understandings how the bonds at the interfaces are formed, as well as the related processes, are of crucial importance by designing and manufacturing of the composites. This review paper describes the interfaces in the magnesium-matrix composites reinforced with different types of SiC, Al_2O_3, and SiO_2, and prepared by different methods.

Key words: magnesium-matrix, reinforcement, composite, interface, reaction product.

Izvleček

Procesi v mejnih območjih med osnovo in utrjevalno sestavino močno vplivajo na lastnosti kompozitov. Osnovna naloga mejnih območij je zagotavljanje trdne povezave med sestavinami kompozita, prav tako morajo biti mejna območja mehansko in termodinamično stabilna. Zato je poznanje načina nastanka povezovanja v mejnih območjih in procesov, ki se zgodijo na njih, zelo pomembno za načrtovanje in izdelavo kompozitov. Pregledni članek opisuje mejna območja v kompozitnih materialih z magnezijevo osnovo, ki so utrjeni z različnimi oblikami SiC, Al_2O_3 in SiO_2 ter izdelani z različnimi postopki.

Ključne besede: magnezijeva osnova, utrjevalna sestavina, kompozit, mejno območje, reakcijski produkt

Introduction

Composites are modern materials, which consist of at least two chemically, physically, and mechanically different materials. The properties of the composites depend on the properties of the matrix and reinforcing phase, shape, fraction, distribution, and orientation of the reinforcing phase, interactions at the matrix/reinforcement interface, processing parameters, and heat treatment conditions. Often, these properties can be predicted, using the rule of mixtures[1]:

$$L_c = L_m V_m + L_r V_r \quad (1)$$

where L_c is the property of the composite (e.g. Young's modulus, density, etc.), V the volume content, and indices m and r indicates the matrix and the reinforcement, respectively.

The interface between matrix and reinforcement is a region with different physical and chemical properties compared to the properties of the composite's constituents. Interface bonding arises from the adhesion of the constituents, which depends on the wettability. In the composites, the wettability is defined as the ability of the liquid matrix to spread over the solid surface of the reinforcement. Physical and chemical processes at the interfaces can strongly influence the mechanical, thermomechanical, and thermodynamic properties of the composites.[1]

Reaction products are formed at the matrix/reinforcement interfaces as a result of the chemical reactions. These interfacial reaction products are usually brittle and could be strongly or weakly bonded to the reinforcement. There exists the critical thickness of the reaction products beyond which the composite properties becomes deteriorated.[2] In the titanium-matrix composites reinforced with SiC fibres, the critical thickness of the reaction products was 1 μm. The mechanical properties of the composite above this value were significantly decreased.[3] Magnesium-matrix composites are promising materials because of their low density and high strength/weight ratio. The specific strength and stiffness of the magnesium-matrix composites should be greater than those of the aluminium-matrix composites. The selection of the ceramic reinforcement (chemical composition, shape, and volume fraction) and magnesium (alloy) matrix can be used to tailor the thermal conductivity of the composites. Particles (p), fibres (f), whiskers (w), and recently also different preforms (e.g. ceramic foam (cf)) are usually used as reinforcements (Figure 1). An advantage of the magnesium, compared to the aluminium, is that it can wet most of the ceramic reinforcement. A disadvantage is its reactivity with the reinforcements. In many cases, the reinforcements are very prone to oxidation. The oxidation behaviour and further reactions could influence interfacial structure and composition, and hence the nature and strength of the interfacial bonding. Undesirable reaction products at or near the interface may lead to loss of the load-bearing ability and thus change the mechanical properties of the composite.[2]

Morphologies of the interfaces

Figure 2 schematically presents distinct types of the matrix/reinforcement interfaces. At the interface type I (Figure 2a), the interfacial reaction products (IRPs) are directly in contact with the reinforcement. For the interface type II (Figure 2b), the interfacial reaction zone (IRZ) consists of two distinct layers. The first layer consists of the IRPs, and this layer is in direct

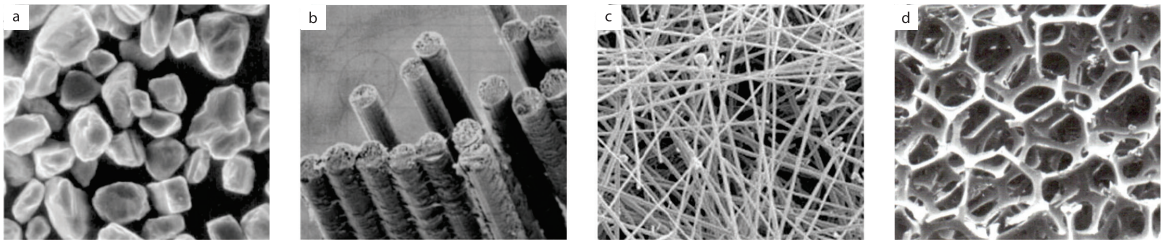

Figure 1: *Shapes of the reinforcements. a) particles (p), b) fibres (f), c) whiskers (w) and d) ceramic foam (cf).*

contact with the matrix. The second layer is in direct contact with the reinforcement, which consists of the matrix that extends along reinforcing surface. Thus, for interface type II the IRPs are not in direct contact with the reinforcement. The interface type III is very clean (Figure 2c) and the IRPs have not even formed at the interface at all.[4] If the matrix does not wet the reinforcement, the cracks and debonding free interfaces are present between them. This interface can be marked as the interface type IV (Figure 2d).

Figure 3 shows the interfaces in the composite with AZ91 matrix reinforced with SiC particles (SiCp), where three kinds of the interfaces were observed.[4] The reasons for existence of these three kinds of the interfaces in the present composite may arise from the conditions during stir-casting, which are very complicated. The friction between the melt and SiCp takes place during stirring and causes shearing during pouring. These actions can cause the formation of the interface III by breaking away the IRPs from the SiCp. However, the IRPs separated from the SiCp and SiCp are often pushed by the freezing front to the last solidified regions, and this leads to the formation of the interface II.[5] When the IRPs do not break away from the SiCp during stirring and pouring, the interface I will be formed.[4]

Types of bonding at the interface

There are two types of bonding at the interface in the metal-matrix composites (MMCs): mechanical bonding and chemical bonding.

Mechanical bonding

Mechanical bonding is formed when the surfaces of the matrix and reinforcement are interconnected, and there are no chemical bonds between them. Interfaces in the MMCs are invariably rough, and the degree of the interfacial roughness increases the strength of the bond. In the MMCs reinforced with the ceramics, the metals generally have a higher coefficient of the thermal expansion than the ceramics. Thus, the metallic matrix in the composite will shrink more than the ceramic reinforcement on the cooling from a high temperature. This will lead to the mechanical gripping of the reinforcement by the matrix even in the absence of any chemical bonding. The matrix infiltrates into the cracks on the reinforcing surface, by the liquid flow or high temperature diffusion, which can also lead to some mechanical bonding.

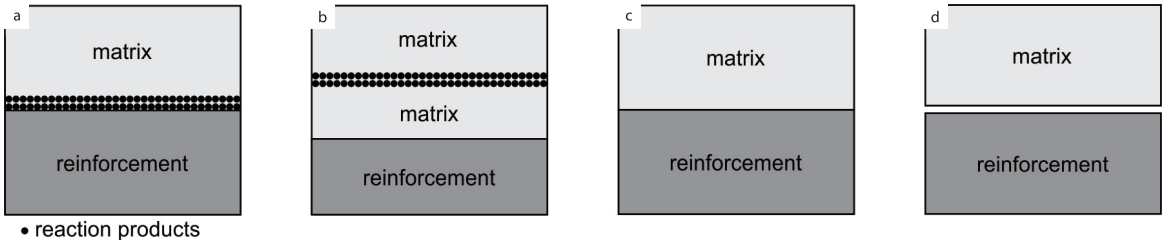

Figure 2: *Schematic representation of the distinct interfaces. a) type I, b) type II, c) type III[4] and d) type IV.*

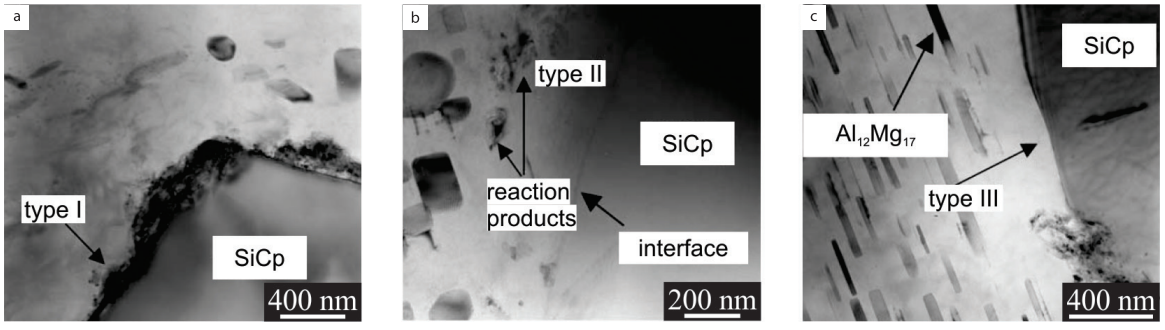

Figure 3: *TEM of the three distinct types of interfaces between AZ91 and SiCp. a) interface I, b) interface II and c) interface III.[4]*

The radial gripping stress, σ_r, can be related to the interfacial shear strength, τ_i, by the equation 2[6]:

$$\tau_i = \mu\sigma_r \quad (2)$$

where μ is the friction coefficient. It usually lies between 0.1 and 0.6. In general, the mechanical bond is a low energy bond vis-à-vis the chemical bond.

Chemical bonding

The metal/ceramics interfaces in the MMCs are generally formed at high temperatures. The diffusion and chemical reaction kinetics are faster at the elevated temperatures. Knowledge of the chemical reaction products and, if possible, their properties are needed. It is, therefore, imperative to understand the thermodynamics and kinetics of the reactions. In this way, the processing can be controlled, and optimum properties obtained. Chemical bonding in the MMCs involves atomic transport by the diffusion, which causes the change of chemical compositions of the constituent phases at the interface. Thus, chemical bonding includes the solid solution and/or chemical compound formation at the interface (Table 1). For the diffusion controlled growth in an infinite diffusion couple with a planar interface, the important relationship is valid[6]:

$$x^2 = Dt \quad (3)$$

where x is the thickness of the reaction layer, D the diffusivity, and t the time. The diffusivity, D, depends on the temperature in an exponential manner[6]:

$$D = D_0 \exp\left(-\frac{\Delta Q}{kT}\right) \quad (4)$$

where D_0 is a pre-exponential constant, ΔQ the activation energy for the rate controlling process, k the Boltzmann's constant, and T the temperature.

Table 1: *Chemical reactions that can take place between magnesium and oxides, carbides, binding agents, and protective gases during the manufacturing of the MMCs*

Phase	Chemical reaction		References
SiO_2	$2Mg_{(l)} + SiO_{2(s)} \rightarrow 2MgO_{(s)} + Si_{(s)}$	{5.1}	4, 7, 8
	$2Mg_{(l)} + 2SiO_{2(s)} \rightarrow Mg_2SiO_{4(s)} + Si_{(s)}$	{5.2}	8
	$4Mg_{(l)} + SiO_{2(s)} \rightarrow 2MgO_{(s)} + Mg_2Si_{(s)}$	{5.3}	2, 8, 9
	$SiO_{2(s)} + MgO_{(s)} \rightarrow MgSiO_{3(s)}$	{5.4}	10
	$SiO_{2(s)} + 2MgO_{(s)} \rightarrow Mg_2SiO_{4(s)}$	{5.5}	10
	$4Al_{(l)} + 3SiO_{2(s)} \rightarrow 2Al_2O_3 + 3Si_{(s)}$	{5.6}	4
	$Mg_{(l)} + 2Al_{(l)} + 2SiO_{2(s)} \rightarrow MgAl_2O_{4(s)} + 2Si_{(s)}$	{5.7}	4, 9
	$4Al_{(l)} + 2MgO_{(s)} + 3SiO_{2(s)} \rightarrow 2MgAl_2O_{4(s)} + 3Si_{(s)}$	{5.8}	4
	$2MgO_{(s)} + 5SiO_{2(s)} + 2Al_2O_{3(s)} (+ C_{(s)}) \rightarrow Mg_2Al_4Si_5O_{18(s)} (+ C_{(s)})$	{5.9}	11
	$2Mg_{(l)} + SiO_{2(s)} (+ C_{(s)}) \rightarrow 2MgO_{(s)} + Si_{(s)} (+ C_{(s)})$	{5.10}	11
	$2Mg_{(l)} + Si_{(s)} \rightarrow Mg_2Si_{(s)}$	{5.11}	4, 7
SiC	$2Mg_{(l)} + SiC_{(s)} \rightarrow Mg_2Si_{(s)} + C_{(s)}$	{5.12}	4
	$4Al_{(l)} + 3C_{(s)} \rightarrow Al_4C_{3(s)}$	{5.13}	4
	$4Al_{(l)} + 3SiC_{(s)} \rightarrow Al_4C_{3(s)} + 3Si_{(s)}$	{5.14}	4
Al_2O_3	$3Mg_{(l)} + Al_2O_{3(s)} \rightarrow 3MgO_{(s)} + 2Al_{(l)}$	{5.15}	2, 12
	$3Mg_{(l)} + 4Al_2O_{3(s)} \rightarrow 3MgAl_2O_{4(s)} + Al_{(l)}$	{5.16}	11
	$MgO_{(s)} + Al_2O_{3(s)} \rightarrow MgAl_2O_{4(s)}$	{5.17}	12
$Al(PO_3)_3$ (binding agent)	$9Mg + Al(PO_3)_3 \rightarrow 9MgO + Al + 3P$	{5.18}	13
N_2 (protective gas)	$3Mg_{(g)} + N_{2(g)} \rightarrow Mg_3N_2$	{5.19}	14
	$Mg_3N_2 + 2Al_{(l)} \rightarrow 2AlN + 3Mg$	{5.20}	14

Interfacial reaction products

The interfaces between magnesium-matrix and reinforcements are not thermodynamically stable thus some interfacial reaction products can be formed (Table 2) as a result of the chemical reactions.

Reactions at the Mg/SiC interface

Magnesium and its alloys reinforced with the SiCp are very interesting because the reinforcement may lead to significant improvement of stiffness and strength.[2] Reaction products at the magnesium/SiC interface depend on the manufacturing method of the composite.

Kaneda and Choh[15] found that the MgO and Mg_2Si reaction products were formed at the pure magnesium/SiCp interface. The feature of this study was previous mixing of the SiO_2 powder infiltration agent with the SiCp reinforcement which is necessary for spontaneous infiltration phenomenon. The Mg–RE3 alloy wets the SiCp well, therefore, in this composite the RE_3Si_2 interfacial reaction products were formed in the form of the needles[16] or thick reaction layer composed of the MgO, and Ce_3Si_2 fine particles.[17] On the other hand, the interfacial reaction products were not observed in the composites with the pure magnesium, Mg–Al5, Mg–Al8, and Mg–Zn6 matrices reinforced with the SiCp and prepared by the melt stir technique[7, 17, 18]. Also, Cao et al.[19, 20] did not find the interfacial reaction products in the Mg–Zn4, and Mg–Zn6 alloys reinforced with the SiC nanoparticles and prepared by the ultrasonic cavitation.

The AZ80/SiCp and AZ91/SiCp interfaces were without reaction products when the composites were prepared by the compocasting. Nevertheless, the particles of the $Al_{12}Mg_{17}$, and Cu_5Zn_8 compounds precipitated on the SiCp[21, 22], indicating that the SiCp acted as nucleation sites. Similarly, the $Mg(Cu, Zn)_2$, and $MgZn_2$ compounds precipitated at the SiCp in the ZC63 - SiCp composite prepared by the melt infiltration into the powder and the melt stir technique. In the ZE63 - SiCp composite, which was prepared by the same procedure, the ZrO_2, and CeO_2 interfacial reaction products were formed.[8] Further Wang et al.[4] found the Al_4C_3, MgO, and Mg_2Si reaction products at the AZ91/SiCp interfaces when the composite was prepared by the melt stir technique. Directly at the reaction layer the carbon was present as a product of a chemical reaction between the magnesium and SiCp {5.12}. Magnesium does not have stable carbides but the aluminium, as an alloying element in the magnesium alloys, reacts with the carbon, and then the Al_4C_3 carbide can arises {5.13}. Also in this case, the SiCp acted as heterogeneous nucleation sites for $Al_{12}Mg_{17}$ and Al_8Mn_5 compounds. When the

Table 2: *Interfacial reaction products formed at the interface between magnesium-matrix and different types of reinforcements*

Matrix	Reinforcement	Interfacial reaction product
Mg	SiCp	MgO, Mg_2Si
Mg-RE3		MgO, RE_3Si_2 or Ce_3Si_2
ZE63		CeO_2, ZrO_2
AZ91		Al_4C_3, MgO, Mg_2Si, $MgAl_2O_4$, AlN
AZ91	SiCw	MgO
Mg	Al_2O_3f	$MgAl_2O_4$, MgO, Mg_2Si
AZ91		MgO
ZE41		MgO
AS21		MgO
AE44		MgO
AZ31	SiO_2cf	MgO, Mg_2Si
AZ61	SiO_2nano-p	MgO, Mg_2Si
AZ31	SiO_2–Al_2O_3cf	$MgAl_2O_4$, $Mg_2Al_4Si_5O_{18}$, Si, Mg_2Si
AZ91	SiC–SiO_2–C–Sicf	MgO, Mg_2Si

AZ91 - SiCp composite was prepared by the ultrasonic cavitation, the interfacial reaction products did not form.[23]

Wu et al.[24] investigated the interfaces between the AZ91 alloy and SiC whiskers (SiCw) in the composite prepared by the squeeze casting. They determined the MgO interfacial reaction products, while Zheng et al.[25] did not find any interfacial reaction products in the same composite. When the $Al(PO_3)_3$ binding agent was added into the SiCw-preform, the MgO interfacial reaction products formed.[13] In the AZ91 - SiC nanoparticles composite prepared by the ultrasonic cavitation, Lan et al.[26] found the Mg_2Si interfacial reaction products, which were broken away from the AZ91/SiC nanoparticles interfaces because of the intensive ultrasonic cavitation.

The MgO, $MgAl_2O_4$, and AlN interfacial reaction products and the $Al_{12}Mg_{17}$ compound were formed in the composite prepared by the melt infiltration of the AZ91 alloy into the premixed powder of the magnesium, aluminium, zinc, and SiC.[27, 28] The AlN reaction layer, which also contained magnesium, is the product of a chemical reaction between the Mg_3N_2 and aluminium {5.20}. The Mg_3N_2 layer around the particles of the powder was formed with reaction {5.19} between the magnesium and nitrogen, which was used as a protective gas.[14] The $MgAl_2O_4$ interfacial reaction product formed in the composites with the aluminium- matrix reinforced with the SiC, and Al_2O_3 when the magnesium content in aluminium was smaller than the mass fraction of Mg 4 % or 2 %.[9, 28] The reactions {5.6}, {5.7}, and {5.8} did not take place because of the large chemical affinity of the magnesium to oxygen and the large content of the magnesium in the magnesium - SiCp composites.

Reactions at the Mg/Al_2O_3 interface

The composites, where the magnesium and its alloys are infiltrated into the reinforcing preform of the Al_2O_3 fibres (Al_2O_3f), are most often prepared by the squeeze casting[29–35]. The preform of the Al_2O_3f contains 3–4 % of the SiO_2 binding agent.

Rehman et al.[36] investigated the matrix/fibre interactions in the composites with the pure magnesium, AZ61, and AZ91 matrices reinforced with the different Al_2O_3f. A few large Mg_2Si particles were found in the pure magnesium reinforced with the δ-Al_2O_3f (Safimax) with standard density. In the case of the reinforcing with the η-Al_2O_3f (Safimax) with low density, the fibres were reduced into the MgO and aluminium {5.15}. The fine MgO interfacial reaction products were observed in the AZ91 - δ-Al_2O_3f (Saffil) composite. It is viable that increasing the aluminium content in the magnesium matrix may reduce the interfacial reactions. Also, Hach[37], Page[38], Hallstedt[39], Trojanová[40] and Skleničkа[41, 42] found the MgO particles at the matrix/fibre interfaces in the Mg - α-Al_2O_3f, Mg - δ-Al_2O_3f (Saffil), ZE41 - α-Al_2O_3f, AS21 - δ-Al_2O_3f (Saffil), and AZ91 - δ-Al_2O_3f (Saffil) composites. The sizes of these particles were higher at the ZE41/α-Al_2O_3f interfaces than at the Mg/α-Al_2O_3f interfaces.[38] They were further increased by increasing casting temperature[43] and longer reaction times.[44] The presence of the spread MgO interfacial reaction layer in the AE44 - Al_2O_3 short-fibres (Saffil) composite has been reported also by Hu et al[45] Besides, they have also found the Al_2RE particles. Similarly to the SiCp also the Al_2O_3f acted as nucleation agents because the β-$Al_{12}Mg_{17}$ compound at the AZ91/δ-Al_2O_3f (Saffil) interfaces and the Al_2Nd, $Mg(Ag)_{12}Nd$, and Mg_3Ag compounds at the QE/δ-Al_2O_3f (Saffil) interfaces were precipitated.[42] Shi et al.[12] found that in the Mg - Al_2O_3f composite the $MgAl_2O_4$ interfacial reaction product was formed, probably with reaction between the MgO and Al_2O_3 {5.17}. It should be noted that, in this study, the magnesium or aluminium powder was added into the preform of the Al_2O_3f and that the reaction time was 4 h at the temperature of 1 123 K. Also in the study of wettability of the α-Al_2O_3f with pure magnesium the $MgAl_2O_4$ interfacial reaction product was found in addition to the MgO.[46–48] This shows that the $MgAl_2O_4$ reaction product is formed after very long reaction times.

Reactions at the Mg/SiO_2 interface

The SiO_2 is seldom used as a reinforcing phase in the form of the particles or in any other form. Most often it is used as a binding agent by the manufacturing of the reinforcing preform of the Al_2O_3 fibres.

The melt is infiltrated into the pores and struts of the ceramic foam at the manufacturing of

interpenetrated phase composites. The Mg_2Si, and MgO reaction products and $Al_{12}Mg_{17}$ compound were formed in the pores and struts of the SiO_2 ceramic foam, which were filled with the AZ31 matrix.[49] Lee et al.[50] incorporated the SiO_2 nanoparticles into the AZ61 matrix by the friction stir processing. The SiO_2 nanoparticles reacted with the magnesium {5.3} and the Mg_2Si, and MgO reaction products were formed.

Reactions at the Mg/SiC + Al_2O_3 + SiO_2 interface

The reinforcing phase can consists of two or more carbides or oxides in the different preforms, e.g. the ceramic foam (cf). Zeschky et al.[11] found at the AZ31/SiO_2–Al_2O_3cf interface the $MgAl_2O_4$ {5.16}, and $Mg_2Al_4Si_5O_{18}$ {5.9} reaction products and the silicon, which further reacted with the magnesium and the Mg_2Si {5.11} was formed. In the AZ91 - oxidized SiC–SiO_2–C–Si(cf) composite, at the interfaces the MgO, and Mg_2Si reaction products, into the struts of the ceramic foam very small content of the MgO, and in the centre of filled pores of the ceramic foam the MgO, Mg_2Si, and γ-$Al_{12}Mg_{17}$ were formed. In the case of reinforcing the AZ91 matrix with the non-oxidized SiC–SiO_2–C–Sicf, the cracks and debonding free interfaces were obtained between the metal and ceramic skeleton.[10]

Conclusions

During the manufacturing of the magnesium-matrix composites, the strong bonding between the matrix and reinforcement, without the reaction products at the interfaces should be attained. However, most of the observed interfaces in the magnesium-matrix composites were covered with the interfacial reaction products. This means that the systems magnesium alloy - reinforcement (SiC, Al_2O_3, and SiO_2) were thermodynamically unstable. The main interfacial reaction products were the MgO, and Mg_2Si. Their size increased with increasing casting temperature and longer reaction time while the increasing the aluminium content into the magnesium matrix reduced the interfacial reactions. The types of the interfacial reaction products were also depended upon the manufacturing method. Therefore, in order to obtain adequate properties of the magnesium-matrix composites, it is necessary to choose the appropriate combination of the constituents and suitable manufacturing process.

Acknowledgement

This work was partly financed by the Slovenian Research Agency (ARRS), projects 1000-09-310152, and L2-2269.

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Review paper

Received: November 23, 2013
Accepted: December 10, 2013

Interdisciplinary research of museum objects: practical experience with various analytical methods

Interdisciplinarne raziskave muzejskih predmetov: praktične izkušnje z različnimi analitskimi metodami

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Abstract

Analytical investigations of museum objects can provide entirely new insights into historical artifacts and ancient technologies. Museum curators and conservators have long since recognized the value of interdisciplinary research. Collaboration with experts versed in technical and material analyses often yields highly encouraging results, uncovering new layers of information that could not be derived otherwise with a traditional museum approach. However, interdisciplinary research of historical artifacts presents serious challenges that may not seem readily apparent at first. In order to obtain optimal results, common ground must be found between the museum curator and conservator on the one hand and the scientific analysts on the other hand. The following paper examines some examples of recent research collaboration carried out on behalf of the National Museum of Slovenia, with an emphasis on metal artifacts and particularly arms and armour. Various analytical methods are discussed based on practical examples, as well as their potentials and limitations. It is hoped that the overview will help promote further interdisciplinary cooperation and possibly contribute toward establishing common standards for future analytical work on museum objects.

Key words: museums, historical artifacts, material analyses, arms and armour, research methodology

Izvleček

Naravoslovne analize lahko odprejo povsem nov vpogled v muzejske predmete in stare tehnologije. Muzejski kustosi in konservatorji se že dolgo zavedajo pomena interdisciplinarnih raziskav. Sodelovanje s specialisti naravoslovnih in tehniških ved pogosto prispeva zelo pozitivne rezultate, saj lahko razkrije popolnoma nove ravni podatkov, do katerih se ne bi mogli dokopati zgolj s tradicionalnim muzejskim načinom. Interdisciplinarne raziskave pa pomenijo tudi svojevrsten izziv, čeprav se tega marsikdaj niti ne zavedamo. Do zares koristnih izsledkov lahko privedejo šele, če muzejskemu kustosu in konservatorju uspe najti skupni jezik s predstavniki naravoslovnih oz. tehniških ved. V prispevku povzemamo nekaj primerov raziskovalnega sodelovanja, ki smo ga v zadnjih letih izvedli pod okriljem Narodnega muzeja Slovenije – s poudarkom na kovinskih predmetih oz. še posebej orožju in bojni opremi. V diskusiji na podlagi praktičnih izkušenj predstavljamo različne analitske metode, ob tem pa opozarjamo na njihove možnosti in pomanjkljivosti. Upamo, da bo takšen pregled pripomogel k nadaljnji krepitvi interdisciplinarnega sodelovanja, morda pa lahko spodbudi tudi k vzpostavitvi splošnih standardov za analitske raziskave muzejskih predmetov v prihodnje.

Ključne besede: muzeji, zgodovinski predmeti, naravoslovne analize, orožje in bojna oprema, raziskovalna metodologija

Introduction

Since their inception, museums have become much more than mere keepers of historical heritage. Their responsibilities have grown increasingly diverse during the last century, but among the most important remains undoubtedly in-depth scholarly research of historical artifacts and material culture.

Museum curators and theorists have long been aware of the fact that every museum object, even one seemingly of little note, represents a unique source of information. Tapping the full information potential of a particular museum object and placing it within a telling historical context is therefore the curator's primary goal. How to achieve that goal in practice – and by what means – remains a matter of discussion, though.[1, 2]

The traditional museum approach is focused on establishing a datation and typology of the historical artifact, relying mostly on the curator's basic training in (art) history, archaeology, ethnology or some other related field of study. Nonetheless, the curator usually lacks the knowledge and equipment required for in-depth analyses of the more technological aspects of the object at hand, such as its workmanship and materials. To some degree, the curator may receive welcome assistance by the museum conservator. However, only systematic scientific and technical analyses of historical artifacts carried out by properly trained specialists can reveal the full scope of their composition, methods of manufacture and material properties.[3]

It is no surprise that such interdisciplinary collaboration has become standard during the past decades. Yet it should not be taken for granted. In most museums, few – if any – formal standards exist specifying how such work is to be carried out and on what methodological ground. For the most part, these considerations are left entirely to the judgement of the respective curator, as well as to the goodwill and experience of analytical experts employed for the examination of a particular historical material.

The purpose of this paper is to present a brief overview of some recent collaborative efforts conducted on behalf of the National Museum of Slovenia (Narodni muzej Slovenije), with an emphasis on the author's experience related mostly to his work as the curator of the arms and armour collection. The strengths and weaknesses of various research methods – used primarily on metal objects – are outlined, pointing out some of the crucial challenges encountered during practical work.

Hopefully, this experience will stimulate even greater interest in analytical research among museum curators employed in various institutions. Moreover, it may help to familiarize specialists in scientific and technical branches with some common demands and issues pertaining to the research of historical artifacts. At any rate, this contribution may be seen as an attempt toward establishing common research standards for future analytical work on museum objects – something that remains lacking to this day not only in Slovenia, but in many other countries across the globe.

Evaluation of a museum object

Determining the authenticity of an antique, its date and place of manufacture is often a demanding task that requires a good deal of knowledge and experience. It is not a process set in stone. In fact, it is not something normally taught at a formal level either. Rather, it is a complex skill refined by the individual over the course of time as an on-the-job learning process based on interaction with antiques and experienced colleagues who may be able to pass on valuable knowledge first-hand.[4]

A museum curator generally begins by visually inspecting the studied object as a whole and establishing a preliminary typology. A comparison to other similar, reliably dated objects with a solid provenance will usually allow the curator to establish at least a rough chronology and place of manufacture. Comparing the material already in the museum's collections and documentation database is likely going to be the first step. Also, specialist literature, museum catalogues and other scholarly publications will be consulted to narrow down the search pattern as far as possible.

If the object conforms well to the comparative material it should be relatively easy to place it within a widely accepted typology. However, a

detailed examination will be necessary to determine whether the object is actually genuine or fake, whether it has been restored to any considerable degree or modified during the course of its working life.[5]

In order to answer the above questions, it is necessary to pay particular attention to the materials and workmanship. Again, a detailed visual examination will be used to check whether the object is made of historically appropriate or "period" materials. Intact surface patina may already point out quite reliably whether the artifact is authentic or a modern fake, perhaps artificially aged to give the impression of an original object. Closer inspection of the surface, possibly under magnification with a loupe or microscope, may also reveal the tell-tale traces of workmanship methods and tools used in the process – forging, stamping, welding, soldering, grinding etc.

Depending on the individual's knowledge and expertise, this traditional approach may yield excellent results. However, its success relies entirely on the curator's knowledge of historical materials and craftsmanship. In a typical history museum the curator is usually an historian, art historian, archaeologist or ethnologist by profession. Although a university degree in one of these fields may prepare the future curator well for most aspects of his trade it does not by itself provide an effective foundation for the advanced study of museum objects in terms of their workmanship.

It is no surprise that the museum curator often works in close tandem with the conservator, a specialist trained in cleaning and preserving antique objects. Through their work, conservators invariably become intimately acquainted with museum objects on their technological level. The conservator's formal background – which may include training in woodworking, metalworking, painting, chemistry, goldsmithing, engineering etc. – can assist the curator greatly in the interpretation of museum objects. Nonetheless, even a seasoned conservator might lack the skills and tools required to make a sound evaluation of the workmanship and materials present in a museum object. Fortunately, this deficiency may be addressed by consulting outside specialists, whose assistance can prove to be an invaluable asset.

Advanced methods and analytical techniques

During the past decades, interdisciplinary work has become increasingly popular in museums. Usually, this involves combining the skills of museum curators and conservators with chemists, engineers, metallurgists and other specialists versed in scientific analytical methods.[6]

An interdisciplinary approach toward studying museum objects can reveal a surprising amount of information otherwise inaccessible to the curator. Properly planned and conducted analyses may answer how a particular object was made, what sort of technology was available in a given historical period, how well the craftsmen mastered their techniques and how their products may have performed in practice. Fakes can be exposed, old restorations and additions identified. Additionally, the analyses may suggest whether a particular method of conservation works well in the long run or whether it should be replaced by a more appropriate technique.

However, such research is also fraught with pitfalls. Since museum objects are by definition precious and irreplaceable, proper analytical methods must be selected in the first place. Nondestructive and noninvasive techniques are generally preferred. Physical removal of samples is often impossible, especially in the case of well preserved antiques, as it would cause irrepairable harm to a sensitive object of great historical value.

Even though interdisciplinary research has become downright fashionable in recent years, it does impose new burdens on both the museum personnel and outside specialists in technical and applied sciences. Quite often, the two sides are initially somewhat incompatible in their methodology and expectations. Hence a considerable mutual effort is required to bridge the gap between their areas of expertise.

Museum curators are often hampered by a general lack of familiarity with scientific analytical methods and technology. An average (art) historian, ethnologist or archaeologist has little to no formal background in material sciences – and possibly little inclination to study the more technical aspects of material culture as represented by museum objects. Under such circum-

stances there may be little desire to carry out any ambitious analytical research in the first place. Sometimes, this is further compounded by an apprehensive attitude toward any sort of technical analyses due to fear – realistically founded or merely perceived – of damaging an historical artifact.

A typical chemist, engineer or metallurgist on the other hand may be well versed in their trade, but this usually involves working with modern materials and technologies. Museum objects are generally products of ancient – and today obsolete – craftsmanship. Many techniques developed and perfected by old crafstmen are poorly understood. A modern expert familiar only with industrial manufacturing methods may struggle with the interpretation of analyses carried out on museum objects, which were the product of a very different age. Also, many analytical techniques taken for granted in the industry may be completely inapplicable to sensitive museum objects. For instance, an intact medieval sword blade cannot be simply sawed in half to examine its cross-section under a microscope.

Great care must be taken to realistically assess whether a particular museum object is suitable for analytical research and what method would yield the best results considering all the constraints and restrictions inherent in dealing with historical artifacts. Perhaps even more importantly, the interdisciplinary research team must first define clear goals of their work – what is the purpose of the attempted analyses, what answers the museum curator is looking for, what methods are available to provide optimal results with a minimum of irreversible effects to the examined objects and how the interpretation of the analyses is going to be of actual benefit to the study of historical heritage – perhaps through publishing the findings in a scholarly paper, developing a new method of conservation, determining the authenticity of a spurious object etc. Unless these issues are addressed beforehand, there is a real danger of carrying out analytical research merely for its own sake – with little positive impact in the long run.

Material analyses at the National museum of Slovenia

The National Museum of Slovenia, founded in 1821, is the oldest public museum and indeed one of the very oldest scientific institutions in Slovenia. Based in the capital city of Ljubljana, it houses some 300 000 objects ranging from prehistory to the contemporary period. As the leading state institution of its kind, the National Museum of Slovenia has a comparatively long history of interdisciplinary scientific research.[7, 8] During the last decades, some basic analytical methods have been carried out in-house, mostly by specialists employed at the Department of Conservation and Restoration. These methods rely mainly on microscopic examinations and XRF analyses. Further analytical work has been carried out in cooperation with other scientific institutions, such as the Jožef Stefan Institute and various faculties of the University of Ljubljana.[9, 10]

The stimulus for analytical research at the Museum is generally two-fold. Most of the basic analyses are carried out on demand of the musem conservators to investigate the material composition of museum objects. In this respect, basic material analyses have become an indispensable tool at the Department of Conservation and Restoration, allowing the conservators to select the most appropriate method of treatment for the particular object. The results of the analyses are also of direct use to the curators, providing a solid identification of historical artifacts and sometimes detecting fakes or later restorations.

More ambitious research is generally planned and supervised by individual curators who specialize in a particular field of study and rely on analytical data to establish a more reliable identification of selected objects, determine their exact age and origin through comparative material and databases, reveal details of their workmanship etc. Since such goals usually require the assistance of an outside specialist or institution, obtaining proper financial support is not easy – especially with the great economic recession in recent years. The Museum's funds have been consistently inadequate for large-scale scientific undertakings, making the struggle for additional resources – research grants,

projects and programs – all the more vital. However, it has also been possible to carry out a sizeable amount of interdisciplinary work through the generous support of other Slovenian public institutions and even private enterprises that have made their resources available to the Musem in joint cooperation on a few particularly interesting or unique challenges.

Some recent examples sorted by methodology

Light microscopy

Detailed visual examination is the first obvious step toward studying any museum object. A hand-held or head-mounted magnifying glass, usually between 5× to 10× magnification, is a highly practical tool. It can already reveal a number of details that cannot be distinguished clearly with the naked eye. The examination is generally focused on crucial details, such as stamps, inscriptions, etching or surface decoration. However, a specialist familiar with historical manufacturing techniques can also detect traces of tools, machining processes and other evidence of workmanship on the surface of the object.

As an inexpensive, easily available and entirely nondestructive method even better results can be obtained with a full-sized microscope. A portable or bench-mounted stereo zoom binocular microscope is ideal for the task. Lower ranges of magnification (10–100-times) are sufficient to observe such details on metal objects. Obviously, greater magnifications are needed for examining properly prepared metallographic samples and identifying textile fibres or organic materials such as bone, antler and ivory.[11, 12]

In museum work, the success of basic light microscopy as a means of identifying workmanship methods and identifying materials is dependent on the operator's skill level and experience. It allows an experienced museum curator or conservator to spot tool marks, traces of machining, welded, brazed or soldered joints, riveting, etching, gilding and other decoration techniques. A systematic visual examination of such details can determine whether the workmanship is consistent with the supposed age of the artifact, whether it was made by hand or machine and if any parts were subjected to a later repair or modification (Figure 1).

Figure 1: *Macro photograph of an old repair – details of riveting and brazing on a 16th century sword blade. (Photo: T. Lazar)*

At the National Museum of Slovenia light microscopy is carried out in-house regularly during conservation treatment. It has been used with effect to identify textile fibers and organic materials. In recent years, light microscopy has been used to investigate an interesting armoured glove – a mail mitten of a type found in several museum collections in the Balkans and identified as late-medieval Ottoman hand defence. However, a close-up identification of the glove has revealed that the metal links were machine-made, as demonstrated by identical wear marks repeated on all the links analysed (Figure 2).[13]

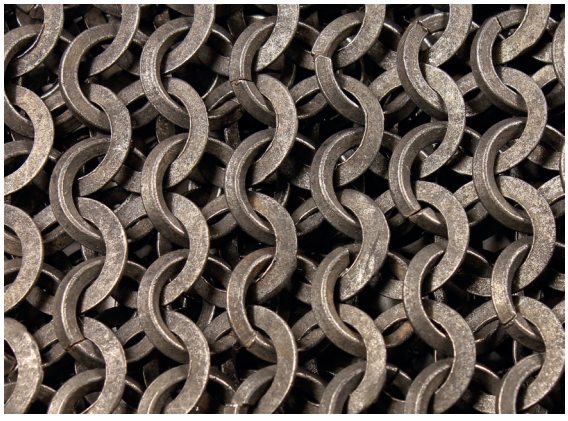

Figure 2: *A detailed examination of a 19th century butcher's mail glove shows discernible tool and wear marks. (Photo: T. Lazar)*

Scanning electron microscopy (SEM)

A complementary method, SEM requires considerably more advanced equipment often inaccessible to the museum curator. In practice, its uses are similar to light microscopy – most notably, microstructure analyses. Also, semi-quantitative composition analyses may be performed with energy dispersive X-ray spectroscopy (EDS) or wavelength-dispersive spectroscopy (WDS). Typically, such analyses cover a surface area of approximately 10 mm in diameter and are restricted to a depth of a few ten µm. The method is particularly useful when dealing with microscopic samples. Still, generally this requires at least a minimally invasive approach, which negatively affects the integrity of the object.[14]

Ultraviolet fluorescence (UVF)

A technique often used in forensic research, UVF has had a long history in art conservation. UV lighting, most commonly in the spectre between 300 nm and 400 nm, creates a highly visible contrast between the original and recent layers of materials applied to the surface. This makes it an ideal research tool for analysing paintings and artwork, where UVF can be used to identify later restorations or additions to the original surface.

Figure 3: *UV photography of a miniature suit of armour easily identifies various layers of varnish. (Photo: Andrej Hirci)*

The method itself is relatively straight-forward and does not require particularly complex equipment. Its use seems to be primarily restricted to art galleries, but it is really much more versatile and can be applied with good effect on historical collections as well. Recently, UVF has been used to analyse two miniature suits of armour from the late 19th century kept at the National Museum of Slovenia. UV photographs have shown very distinctly the difference between the original surface and all the later conservation treatments as well as attempts at more extensive restoration (Figure 3).

Energy dispersive X-ray fluorescence (EDXRF)

In-house EDXRF analyses have been performed at the Museum regularly since 1999, when a custom-made EDXRF apparatus was acquired from the Jožef Stefan Institute. It has become indispensable to the Museum's curators and conservators.

Initially, EDXRF analyses have been used primarily as a quick, noninvasive means of roughly identifying the object's composition. However, the increasingly more sophisticated equipment and software developed by the Jožef Stefan Institute have opened up new possibilities – at this point, much more accurate quantitative analyses of material composition have become possible. For instance, the current PDZ-01 device developed at the Institute can provide a quantitative analysis of elements from Al to U with an inherent uncertainty of some 5–10 %, depending on the homogeneity of the sample. The beam diameter covers an area of roughly 0.9 cm in diameter, reaching to a depth of some 10–100 µm depending on the composition of the object. Furthermore, specialized methods can be used, such as measuring the thickness of film applied to the surface of the object (e.g. gilding, tinning, electroplating).

Particularly good results have been obtained on objects made of nonferrous metals, such as bronze or brass, gold, silver and tin alloys.

Advanced EDXRF analyses can provide relatively accurate information on their composition and help identify the alloying elements, even if present in minute quantities. The method is somewhat less useful for iron or steel, as it cannot determine their carbon content. Nonetheless, most other common alloying elements can be detected and quantified.[15–17]

At any rate, it is generally necessary to take a number of readings on each examined object in order to arrive at statistically reliable average values – obviously depending on the size of the beam as well. This is particularly important when dealing with heterogeneous materials whose composition may vary a good deal throughout the object.

The method is entirely nondestructive per se. Due to limited penetration of the beam, the readings are representative only of the microscopic surface layer. If a portable device is used, analyses may be carried out in-situ, even on relatively inaccessible parts. This is an important advantage, as the transport of large or particularly sensitive and valuable museum objects to a research laboratory may be highly impractical and expensive.

Although the analyses require no special surface preparation it is nevertheless important to note that secondary contamination may distort the results. In almost all cases, Ca has been detected on metal objects, most likely due to contamination with dust. The unexplained presence of Cu and Zn on steel or iron artifacts has also caused considerable confusion. During the recent in-depth analyses of a 15th century sword blade it has been proved with additional testing that the readings of Cu and Zn must be attributed to later contamination during conservation treatment – in the past, brass brushes were frequently used at the Museum for mechanical cleaning but their application invariably left microscopic residue of brass on the surface (Figure 4).[14]

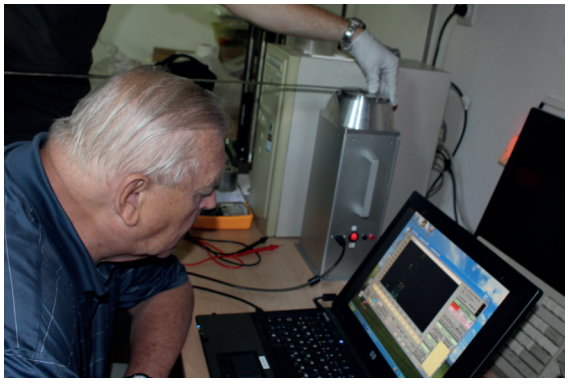

Figure 4: *EDXRF examination of a sword blade. (Photo: N. Nemeček)*

In another instance, As was found on the surface of Indonesian kris daggers – clear evidence of the ritual cleaning process using warangan, a compound containing liquid As. Hence, one must factor in such occurences when dealing with historical artifacts.[18]

Particle-induced X-ray emission

Another nondestructive analytical method with a proven track record, PIXE has been applied quite extensively to the study of paintings and museum artifacts. Largely comparable to EDXRF analyses, it shares many advantages and limitations. PIXE may be used to detect only the presence of elements lighter than Si. Above all, the measurement is limited to the very surface of the object (≈ 10 μm).[19–22]

The application of in-air proton beam of the Tandetron accelerator of the Jožef Stefan Institute has been used to investigate some particularly heterogeneous objects. The tightly focused beam with a surface area in the range of 1 mm^2 is very useful for measuring the composition of isolated inclusions or impurities on the surface of the object. During an investigation of Indonesian kris daggers, the PIXE method has been able to confirm the high Ni content in highly visible silvery patches on the surface. As high quality kris daggers were reportedly made of meteorite steel, the analyses have given new evidence for such practice – albeit only in older blades of particularly good workmanship.[18]

X-ray radiography

Investigations with X-ray radiography have long ago become commonplace in museum work. Especially in the period after World War II the easy availability of X-ray technology has led the Department of Conservation and Restoration of the National Museum of Slovenia to establish regular links with laboratories specialized in technical radiography (Figure 5).

X-ray investigations have been found very useful as a preliminary step prior to conservation treatment, especially when dealing with an

archaeological find or a heavily corroded object with an encrusted surface. Radiography may reveal quite clearly how much of the object's metal core is preserved and whether any additional parts or components remain hidden underneath the layer of corrosion products.
For instance, historical metalwork is frequently ornamented with inlays, engravings or some other means of decoration that might be removed during mechanical cleaning unknowingly. One may be dealing with a composite object, including organic materials. Through X-ray radiography such factors may be discovered noninvasively – as well as the location of rivets, joints, brazing or soldering etc.[23]
Radiographic images can also reveal the complex interior of objects such as sword blades deposited in a sheath or the arrangement of a lock mechanism in an antique crossbow or firearm without the need to dismantle the object completely. Again, such data is invaluable for scholarly study as much as it is of great assistance to museum conservators.[24]
Overall, X-ray radiography is a highly practical method for investigating a broad range of historical artifacts. However, its usefulness is necessarily limited by the thickness of metal. When dealing with particularly large, solid objects or those of composite structure alternative methods may offer better results (Figure 6).

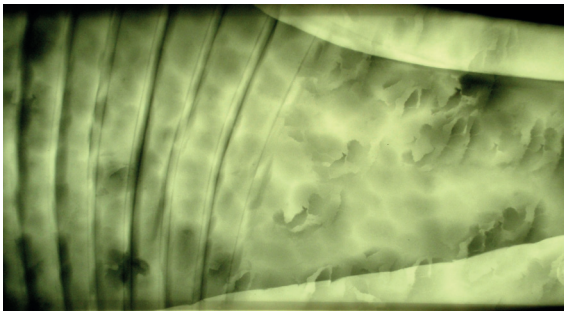

Figure 5: *X-ray radiography of a 16th century breastplate. (Photo: M. Žgavec, B. Zorc)*

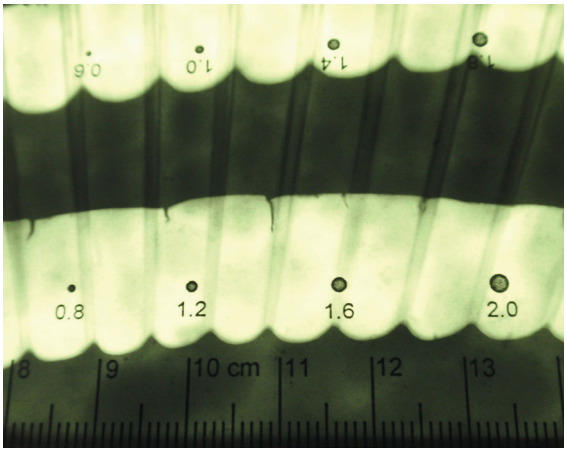

Figure 6: *Apart from detecting invisible details, inclusions and various internal flaws, a radiographic image may also be used to gauge the thickness of metal such as in the case of a skirt belonging to a 16th century suit of armour. (Photo: M. Žgavec, B. Zorc)*

Neutron radiography

As a complementary nondestructive method, neutron radiography offers useful information otherwise impossible to obtain by X-ray imaging. Many of the most commonly used metals, such as Fe, Cu, Sb, Zn or Pb, as well as earthenware or glass, are penetrated easily by neutrons – in contrast to light organic materials.
Therefore, neutron radiography can be used to detect the presence of organic materials hidden underneath a metallic surface. It is particularly useful when dealing with composite objects containing wood, leather, textile or plant fibres.[25, 26]

Ir- and Co-radiography

Objects made of thicker, solid metal that cannot be penetrated efficiently by X-ray may be examined successfully by using a radioactive isotope such as Ir-192 or Co-60. Such specialized radiographic equipment is not easily obtainable, being limited to large-scale industrial production and testing. However, Ir- or Co-radiography can be used as a particularly valuable means for analysing the composition and manufacturing techniques of large museum objects such as cannon barrels (Figure 7).[27]
Recently, the two oldest surviving medieval artillery pieces in Slovenia have been subjected to extensive research. Of greatest importance were the radiographic analyses carried out with an Ir-192 isotope placed inside the barrels and a more powerful Co-60 source positioned vertically above the guns. Due to the considerable thickness of metal (over 10 cm in the thickest sections) a very long exposure time was necessary – up to 12 h. The images recorded on photographic film show clearly the complex construction of late-medieval gun barrels made of wrought iron (Figure 8).[28]

Figure 7: *Preparation for Ir-radiography of a late medieval cannon. (Photo: T. Lazar)*

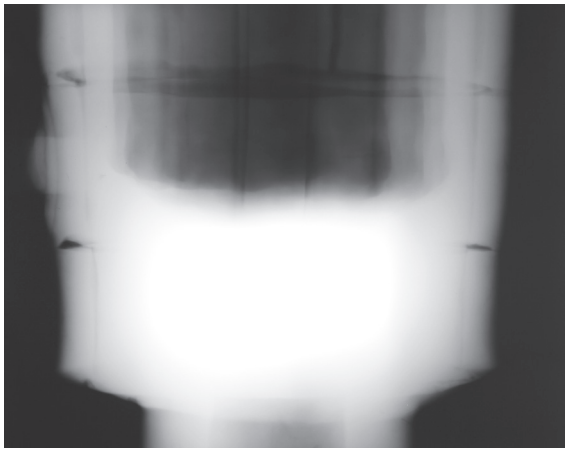

Figure 8: *Co-60 radiography of a 15th century gun or bombard. (Photo: A. Hudej)*

Metallography

Metallographic analyses represent a technically simple and inexpensive, but altogether exceptionally useful method for determining the microstructure and material properties of a metal object. They are particularly valuable for analysing steel tools or weapons. In simplest terms, they require the removal of a sample – which may be quite small or nearly microscopic – that is then ground, polished, etched and examined under a microscope.[29]

Due to its destructive method, metallography is sometimes considered impractical in museum work. In some cases, especially when dealing with fragmentary objects or considerably damaged archaeological finds, it may be relatively easy to detach small flakes without affecting the overall integrity of the object to any major degree. On historical armour, bits of metal can be cut relatively unobtrusively from the inside of rolled or turned edges etc. Otherwise, reasonably inconspicuos removal of samples may be impossible. At best, one might decide to polish and etch very small sections of the surface and examine them in-situ on an inverted microscope. However, such an approach enables the researcher to determine merely the micostructure of the very surface – that may not be representative of the microstructures deeper within the core of the object (Figure 9).[30]

It is important to note that most functional steel objects of the preindustrial era, such as tools, weapons or armour, exhibit a highly complex internal structure. In the first place, the metal may be of highly heterogeneous composition, containing large quanities of slag and impurities. Quite often, the outer surface, especially cutting edges, is carburized or made of a relatively harder steel with a higher carbon content. However, the core may be much softer, possibly forged of wrought iron welded to an outer jacket of higher quality steel. Therefore, it is highly desirable to remove samples from various sections of the object in order to obtain a clear picture of its workmanship and arrive at statistically acceptable values.[31–38]

Within its limitations, metallography offers potentials so far unrivalled by any standard noninvasive analytical method. It may be used to determine the quality of materials used as well as the heat treatment or cold working techniques used during its manufacture. The latter may be used to asses the maker's degree of technological skill and capabilities. In case of historical arms and armour, various techniques of heat treatment can provide a unique

Figure 9: *Careful removal of a small sample from a late medieval brestplate. (Photo: T. Lazar)*

fingerprint, helping identify unknown specimen and ascribing them to various workshops known for a trademark manufacturing procedure (Figure 10).[14, 39, 40]

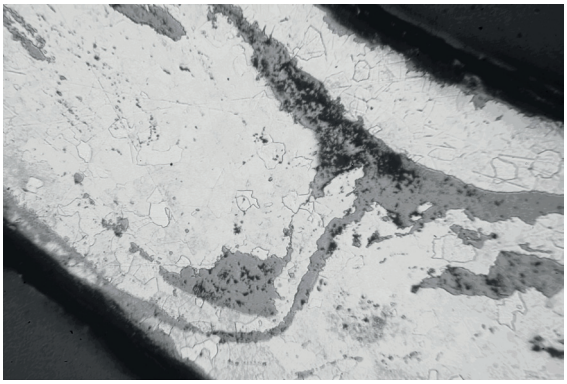

Figure 10: *Metallographic examination of a welded iron link from a medieval mail armour. (Photo: E. Wood)*

Hardness testing

Several methods of hardness measurements exist. Perhaps the one most commonly used on historical artifacts made of iron and steel is the Vickers pyramid method. Vickers microhardness testing is a minimally invasive technique, leaving only a microscopic indentation on the surface of the object. For that rason, it is highly versatile and can be employed on any metal object as long as the appropriate equipment is used and the surface on the measuring location is sufficiently smooth, even and free of corrosion products (Figure 11).

A portable hardness tester, generally operating on the UCI principle, is a highly versatile and accurate analytical tool. As a stand-alone method, hardness testing is of limited value as the results provide only a general indication of the object's material composition and heat treatment. However, in combination with metallographic analyses, systematic hardness testing can be used to assess the uniformity of the object's microstructure and the quality of heat treatment (Figure 12).[14, 18, 30–34]

On the other hand, hardness measurements as well as metallographic analyses can reveal the true microstructure and workmanship of a historical artifact only insofar as it has not been altered during its later life. During the preliminary hardness testing of a number of medieval swords from the National Museum of Slovenia it has been found that many of their blades were surprisingly soft, ranging around 100 HV 0.2. The readings seemed altogether incompatible with the fine workmanship of the specimen, which were clearly well made weapons that one would expect to have been heat treated according to the best capabilities of the contemporary bladesmiths.

However, the surprisingly low values might be explained by an unexpected twist. During the late 19th and early 20th century, conservators would frequently treat historical steel objects by heating them to red heat (around 900 °C), then cleaning them in an acid bath. Such a treatment would invariably anneal the object and destroy its original microstructure. Although very little conservation documentation from the period exists at the National Museum of Slovenia, there is nevertheless clear evidence that in 1906 such an approach was used at least on two early medieval swords from Kranj, and possibly more specimen in later years.[41]

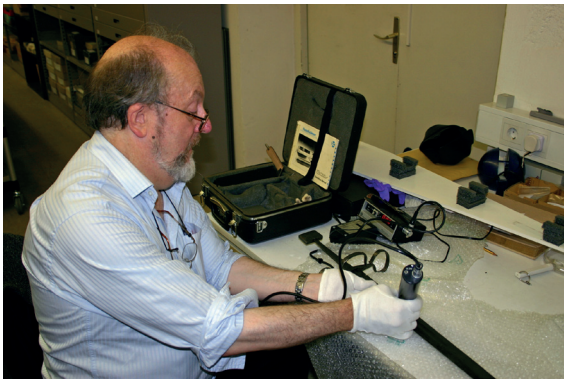

Figure 11: *Investigating a 15th century sword blade with a portable hardness tester. (Photo: T. Lazar)*

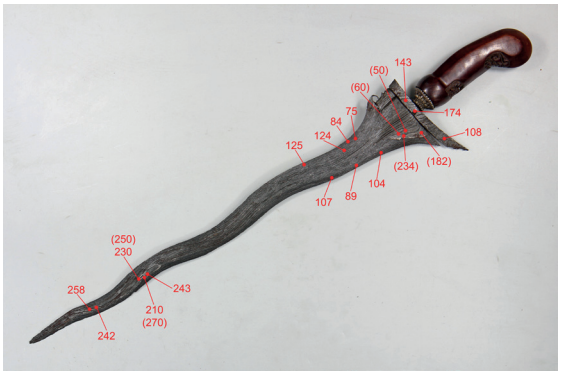

Figure 12: *Hardness measurements on an Indonesian kris dagger. (Photo: T. Lazar)*

Differential scanning calorimetry (DSC)

Thermal analysis of metal samples is destructive insofar as small samples need to be removed from the object, then heated to a very high temperature. During the process, phase transitions can be observed, thus providing an exact identification of the metal's material properties.[42]

During our research at the National Museum of Slovenia, DSC has been tested for the first time during the analyses of a broken late-15th century sword or Messer. The results have shown the method to be of considerable value, showing great potential for further work whenever samples can be removed with a minimum risk of affecting the object's integrity.[14]

Conclusions

Close collaboration between museums and specialists in technical and applied sciences has proved its benefits time and again. Thanks to such interdisciplinary research, our knowledge of ancient technologies and craftsmanship techniques has increased exponentially. Nonetheless, it is crucial to understand that historical artifacts present particular challenges, which the research team must be aware of beforehand.

Objects of cultural heritage are bound by highly specific standards of preservation. In practice, this may rule out a number of analytical methods that might yield the best results in theory but are simply inapplicable due to their destructiveness. Especially when dealing with well preserved artifacts of great value, nondestructive methods may be the only realistic option despite their possible shortcomings. Highly invasive procedures, such as removing large sections of material or polishing extensive surfaces on an object, may cause irrepairable harm to an otherwise unique artifact. For that reason alone, they should be avoided unless absolutely necessary.

A conscious decision may be made to sacrifice a particular object for extensive destructive analyses – such as sawing an object in sections or removing large samples. But such a decision should never be taken lightly. It may be permissible only when dealing with an artifact of no unique value – for example, when a large group of more or less identical specimen is available and the results are expected to justify such drastic measures.

Scientific analyses can provide seemingly extremely exact information. However, the actual value of such information in itself may be quite limited or even misleading unless interpreted in the correct context. For example, metallographic analyses of a medieval sword blade may reliably reveal the microstructure and material properties at the analysed locations. However, these locations may not be representative of the entire blade unless a large number of samples were removed or an alternative method used to check the uniformity of the examined object. Unlike most modern industrial products made of homogeneous materials, historical artifacts tend to vary far more in their material composition and properties.

The interpretation of results may prove to be a highly problematic issue. An analyst whose working experience is limited solely to modern materials and manufacturing techniques may not be able to understand the pitfalls of the great technological gap between the 21st century and the earlier historical periods. A particular historical artifact, such as a pattern-welded blade or armour forged of wrought iron, may have been a technological marvel in its time. Yet purely by today's standards, it could be seen anachronistically as a markedly inferior product. Again, one should not lose track of the technological level of the historical era in question.

It should never be assumed that a particular artifact has not been altered or tampered with during more recent periods. Unless its full history is known and documented, it is quite possible that the object may have been subject to a later repair, modification or aggressive conservation treatment that might have affected its microstructure and material properties.

As much as scientific analytical methods may help with the identification of an historical artifact, the museum curator should be wary of drawing quick conclusions based on limited analytical data. If at all possible, published analyses of similar historical objects should be studied and cross-checked to see if the obtained results are believable or seem out of place.

In case of doubt, it is always advisable to check again for any errors in the analytical process. The limitations of analytical research must be cleared up beforehand. The curator may be under the false impression that a given scientific method will automatically determine the object's age and provenance. In reality, it only provides data that must be compared to other known samples and analyses before any such conclusion can be made. Hence, it is not only worthwile but highly necessary to publish all the analytical results as comprehensively as possible or at least structure them within an internal database to ensure that the work will be of benefit to future research and possibly other research teams as well.

Acknowledgements

The authors must express their gratitude to a number of colleagues who have contributed greatly to the research of collections at the National Museum of Slovenia. Above all, Dr. Marijan Nečemer and Dr. Peter Kump of the Jožef Stefan Institute for their work on nondestructive analyses that have set new standards in Slovenian museums. Special thanks to Dr. Alan Williams – his contribution has been a source of inspiration as well as close collaboration, resulting in interesting joint projects. And last but not least, Dr. Peter Fajfar, the driving force behind recent metallurgical research involving the historical arms and armour at the National Museum of Slovenia, without whose suggestion this paper would not have been published in the first place.

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Professional paper

Received: November 18, 2013
Accepted: December 9, 2013

Variability of chemical composition of metallurgical slags after steel production

Raznolika kemična sestava jeklarskih žlinder

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Abstract

Chemical composition of slags after steel production is variable and depends on: the type of the used charge material, fluxes, refining additives and a used melt technology. Slags contain these elements, which participated in the metallurgical process; they often contain some quantities of heavy metals. For using slags as a secondary material it is very important to know the forms of metals occurrence and their relationship with the slag components. Knowing waste material we can choose a right way of wastes management.

Key words: slags after steel production, chemical composition, heavy metals

Izvleček

Kemična sestava žlinder, ki nastajajo med proizvodnjo jekla, je raznolika. Odvisna je od vložka, žlindrotvornih dodatkov in legur ter tehnologije izdelave. Elementi se vežejo v žlindre med metalurškimi procesi v reaktorju, zato pogosto vsebujejo tudi sledi težkih kovin.
Pri uporabi žlinder kot sekundarnih surovin je zelo pomembno, v kakšni obliki so kovine vezane v žlindri, prav tako pa je potrebno poznati povezavo teh spojin z drugimi komponentami v žlindri. Le tako lahko izberemo pravilno metodo ravnanja z odpadki.

Ključne besede: jeklarske žlindre, kemična sestava, težke kovine

Introduction

Upper Silesia, situated in the southern part of Poland, is one of the best industrialized regions. The beginnings of mining and smelting date here back to the Middle Ages. Besides coal mining, iron and steel industry has become one of the most developed industries in Upper Silesia. But on the other hand, iron and steel industry has become especially problematic, because of considerable amounts of wastes – mainly metallurgical slags. In Poland, there are waste dumps left after industrial establishments' activities from previous centuries. At present there are propositions to apply slag for the production of road aggregate, aggregate for the production of concrete mixtures, there are also attempts to return slags to metallurgical processes, such an activity is popular not only in Poland.[1, 2]

Before using slags as a secondary material it is very important to know their chemical composition, forms of metals occurrence, the resistance of minerals to weathering processes and in which conditions metals are liberated from slag components.[3] This knowledge will be useful in economic activities because utilization of slags should be economically viable and ecologically safe for the environment.

Research methods

To show the chemical composition of the slag the following research methods were used: INAA – Instrumental Neutron Activation Analysis and, TD-ICP – Total Digestion with Inductively Coupled Plasma. The researches were done in Activation Laboratories Ltd. – Actlabs in Canada. The microscopy analysis in transmitted light (on thin plates) was carried out in the Institute of Applied Geology of the Faculty of Mining and Geology of the Silesian University of Technology using the microscope Axioplan 2 of the firm ZEISS for the research in transmitted light and reflected one. The research with the application of scanning microscopy was carried out in the Scanning Microscopy Laboratory of Biological and Geological Sciences of the Department of Biology and Earth Sciences of the Jagiellonian University (Laboratory in the Institute of Geological Sciences). For the research a scanning electron microscope with field emission Hitachi S-4700, furnished with the EDS analysis system (energy dispersion spectrometry) Noran Vantage was applied.

Characteristic of waste material

A number of samples was taken from two dumps located in Upper Silesia (Figure 1).

The first examined dump is the remainder of the activities of steel works, which started working as a production plant on 25th October 1802. But at this moment that steelworks is already closed, after its work only a waste dump has remained. The waste material collected on the dump was stored up not selectively and it contains slag from smelting processes, raw slag from other processes and casting slag. For that reason in the part of the dump remaining after exploitation four strata characterized by a different colour (grey and brown) can be recognized. Vitrified fragments of metallurgical slag can also be seen on the dump. Slags which have

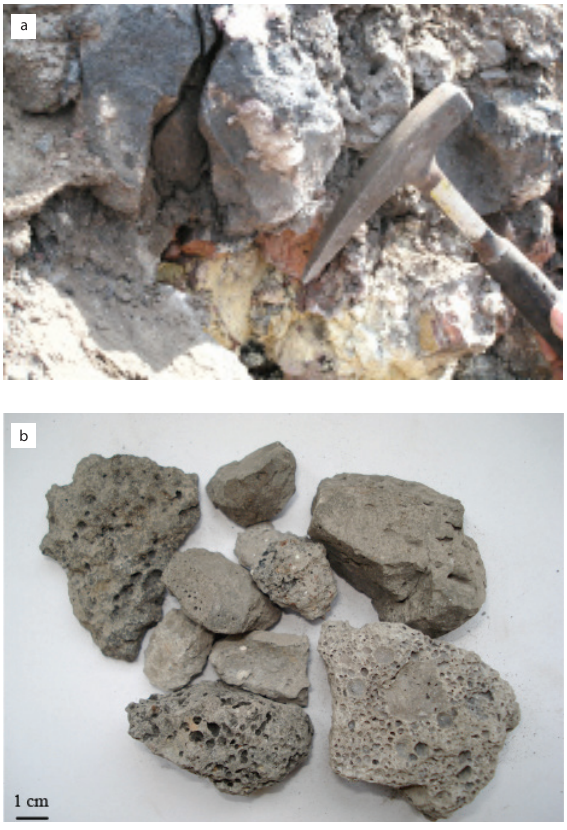


Figure 1: *Slags from the first (a) and the other dump (b).*

been exposed to weathering processes are covered with a white deposit of re-crystallized gypsum and calcite. Currently, the area occupied by the dump is reclaimed. As the material has been stored non-selectively; the studied samples consist of a mixture of slags from various steelmaking processes. At the moment, due to the strong weathering processes, it is difficult to distinguish between different kinds of slags.
The other group of samples was taken from the dump of steel works – one of the biggest iron and steel works in Poland, which started production in 1975. The dump of this steelworks occupies an open-air area of 28 ha. Wastes gathered on the dump represent mainly converter slag. Slags are not weathered; the oldest of them have been gathered on the dump for several years.

Tests results

The following components are present in the chemical composition of wastes:
— non-metals,
— metals,
— and also trace amounts of lanthanides.

Examples of an analysis of the chemical composition of slags from the studied dumps are given below. Concentration of individual elements has been determined in the average and representative sample from each dump (Table 1).
Slag after steel production contains these elements, which participated in the metallurgical process. Therefore, its chemical composition can be determined for the presence of metals such as iron. In the waste material the content of this element should be as low as possible, because this is the determinant of a well-run process of steel production.
Slag from the dump No 1 representing wastes from different steelmaking processes contains the mass fraction w = 11.90 % of iron, while the converter slag from the dump No 2 – 14.20 %. In comparison, based on the research carried out by the author, in the slags from Siemens-Martin process content of iron ranges from w = 4.20 % to 15.90 %[5], while in the wastes from the production of cast steel it has only reached 1.59 %.

Table 1: *Detailed chemical composition of studied slags (examples of characteristic analyses of slags from each dump)*

Element	Unit	Limit of detection	No of dump 1*	2
Ag	µg/g	0.3	1.2	1
Al	%	0.01	4.04	3.19
As	µg/g	0.5	3.3	6.1
Ba	µg/g	50	530	370
Au	ng/g	2	13	< 2
Be	µg/g	1	6	1
Bi	µg/g	2	< 2	< 2
Br	µg/g	0.5	< 0.5	1.1
Ca	%	0.01	12.00	20.00
Cd	µg/g	0.3	0.4	5.4
Co	µg/g	1	36	6
Cr	µg/g	2	214	1180
Cs	µg/g	1	2	< 1
Cu	µg/g	1	837	109
Fe	%	0.01	11.90	14.20
Hf	µg/g	1	3	< 1
Hg	µg/g	1	< 1	< 5
Ir	ng/g	5	< 5	6
K	%	0.01	0.53	0.18
Mg	%	0.01	2.94	3.74
Mn	µg/g	1	14 900	11 400
Mo	µg/g	0.3	< 1	32
Na	%	0.01	0.17	0.13
Ni	µg/g	1	93	0.284
P	%	0.001	0.088	975
Pb	µg/g	3	19	< 15
Rb	µg/g	15	< 15	< 15
S	%	0.01	0.03	0.2
Sb	µg/g	0.1	0.9	48.4
Sc	µg/g	0.1	11.8	2.7
Se	µg/g	3	< 3	< 3
Si	%	0.01	18.64	8.28
Sn	%	0.01	< 0.01	< 0.01
Sr	µg/g	1	232	168
Ta	µg/g	0.5	< 0.5	< 0.5
Th	µg/g	0.2	4	1.7
Ti	%	0.01	0.23	0.16
U	µg/g	0.5	3.9	< 0.5
W	µg/g	1	24	47
Zn	µg/g	1	463	812
La	µg/g	0.5	16.1	7.9
Ce	µg/g	3	28	13
Nd	µg/g	5	7	< 5
Sm	µg/g	0.1	2.2	1.1
Eu	µg/g	0.2	0.6	0.2
Tb	µg/g	0.5	< 0.5	< 0.5
Yb	µg/g	0.2	1.3	0.6
Lu	µg/g	0.05	0.22	0.16
V	µg/g	2	266	686
Y	µg/g	1	26	9

Explanation:
*according to Jonczy 2008[4]

In slags significant amounts of: copper, chromium, manganese and vanadium were also shown. The presence of these elements is connected with the metallurgical process and the type of produced steel. These elements are the additives which improve the properties of steel. In the studied slags special attention is paid to a quite large concentration of manganese (11 400 μg/g and 14 900 μg/g) and chromium (214 μg/g and 1 180 μg/g). A similar situation was observed in the slags from Siemens-Martin process, in which a significant concentration of Mn (20 000 μg/g) and Cr (13 600 μg/g) was noticed.[5]

Slags contain also considerable amounts of zinc. The presence of this element is often associated with the charge material, to which a scrap is added. Slags from the first dump contain 463 μg/g of zinc, while in the slags from the dump No 2 the amount of zinc is increased to 812 μg/g. But the highest concentration of this element was noticed in slags from Siemens-Martin process – 40 500 μg/g.

Slags after iron and steel production usually are characterized by very good technical properties, often compared to properties of natural rocks. At present in Poland, in view of a widely accepted pro-ecological policy, attention has been drawn to the possibility of reusing metallurgical slags, both the slags collected on dumps and the slags generated by ongoing production processes. In this way it will be possible to acquire new materials for example for the production of road aggregate and at the same time to recover the lands previously occupied by dumps.

Therefore, multi-directional research of metallurgical slag should be carried out. Such researches should be applied not only to the determination of the technical properties of slags, but also to the determination of their chemical composition. A very important issue is also to determine the forms of elements occurrence, especially in respect of heavy metals. In the slags metals can form metallic aggregates, their own minerals; on the other hand metals can make substitution in the internal structure of silicates. A considerable amounts of metals are dispersed in glaze and amorphous substance. All these forms of metals occurrences were found in the studied slags (Figures 2–4).

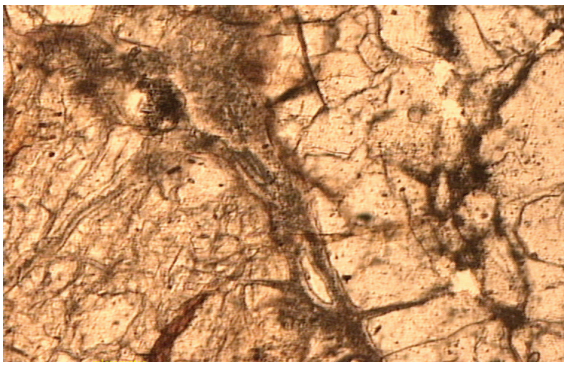


Figure 2: *Metals in cracks of glaze: transmitted light, magnification 100 ×, one nicol.*

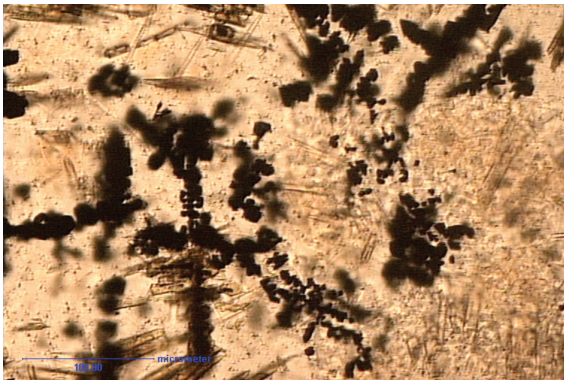


Figure 3: *Magnetite; transmitted light, magnification 200 ×, one nicol*[6].

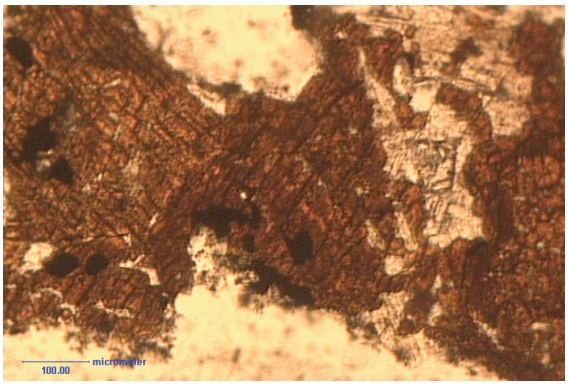


Figure 4: *Inclusions of metal in pyroxenes; transmitted light, magnification 100 ×, one nicol*[7].

Chemical and mineral composition of steel slags is often very diversified. Phases which crystallized in a furnace can be identified with the minerals forming as a result of geological processes. However, its chemical composition is usually much richer than their natural counterparts.

To show it the studies in microarea, from which the chemical composition of the individual slag

components could be determined, are very useful. Varieties of chemical composition of phases, in two microareas of the same sample from the dump No 2, were shown (Figure 5, Tables 2, 3 and Figure 6, Tables 4, 5).

It should be noted that in one sample of the converter slag, in two studied microareas, different phase composition was found. The first microarea is dominated by calcium silicates surrounded by glaze. In the other microarea, oxide phases can be distinguished; they are represented by solutions of mixed oxides of Fe, Mn, Mg and Ca. This shows a very high variability and diversity of mineralogical and chemical composition of the slag. In Tables 3 and 5, the contributions of individual oxides at a given point of analysis calculated to 100 % were shown.

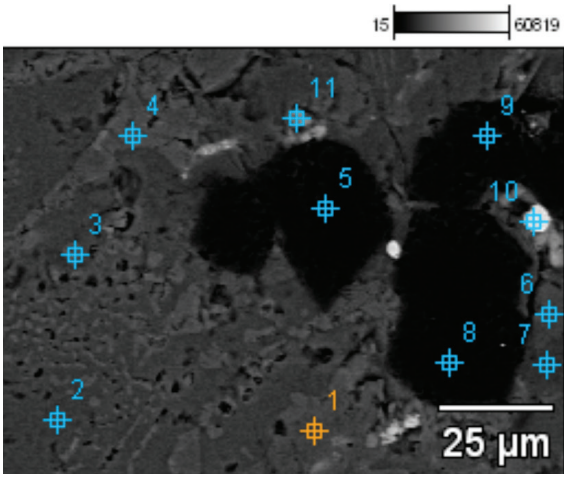


Figure 5: *Microphotography of slag by scanning Microscopy (1) (BSE).*

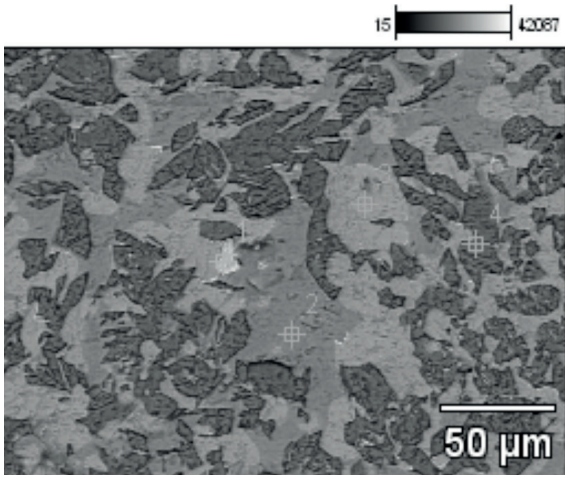


Figure 6: *Microphotography of slag by scanning Microscopy (2) (BSE).*

Table 2: *Chemical composition of slag phases according to Figure 5*

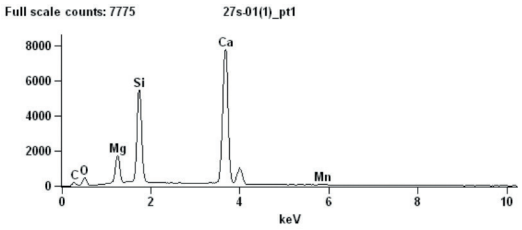
Point of analysis	Oxides [*w*/%]											Σ
	SiO_2	TiO_2	Al_2O_3	FeO	MnO	MgO	CaO	K_2O	V_2O_5	SO_3	P_2O_5	
1	36.19	-	-	-	0.61	11.38	51.83	-	-	-	-	100.01
2	-	-	49.63	-	-	-	50.13	-	-	-	0.24	100.00
3	29.67	-	9.65	-	0.38	4.72	55.59	-	-	-	-	100.01
4	35.39	0.32	-	-	1.05	5.22	58.01	-	-	-	-	99.99
5	0.73	-	-	-	2.65	95.96	0.66	-	-	-	-	100.00
6	35.96	-	-	-	0.66	11.32	52.06	-	-	-	-	100.00
7	21.23	-	35.73	-	0.36	-	42.68	-	-	-	-	100.00
8	0.77	-	-	-	2.84	95.63	0.77	-	-	-	-	100.01
9	0.64	-	-	-	4.51	93.22	1.22	-	-	-	0.42	100.01
11	8.95	2.94	1.11	0.49	6.35	2.65	39.96	0.44	0.03	37.07	-	99.99

Point of analysis	Elements [*w*/%]								Σ
	Si	Cr	Al	Fe	Mn	Mg	Ca	P	
10*	0.20	1.63	0.42	80.66	15.40	0.52	0.98	0.19	100.00

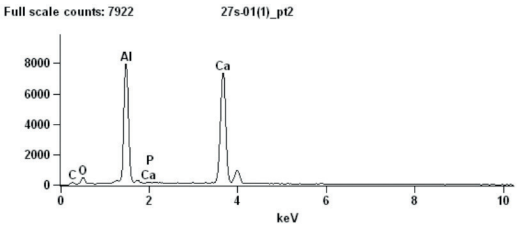
Explanation:
*in the point no 10 oxygen was not determined
Points: 3, 4, 6, 7 – calcium or dicalcium silicates, 2 – calcium aluminate, 5, 8, 9 – periclase, 11 – glaze

Table 3: *EDS spectrums (Scanning Microscopy) according to Figure 5*

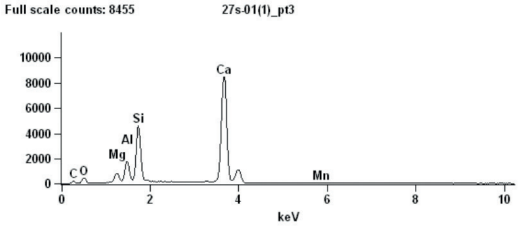
Point 1



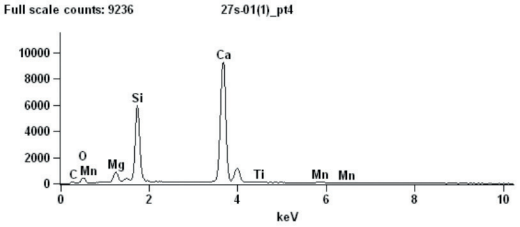
Point 2



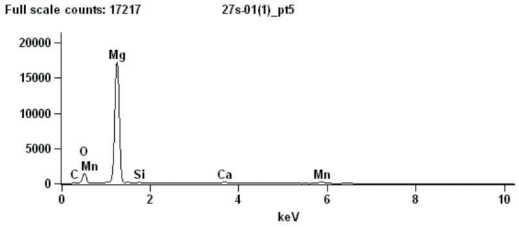
Point 3



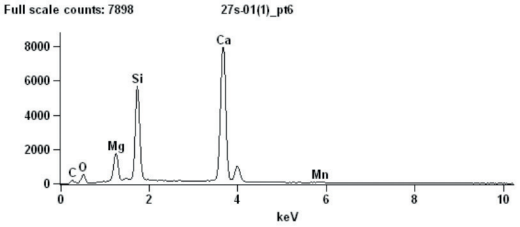
Point 4



Point 5



Point 6



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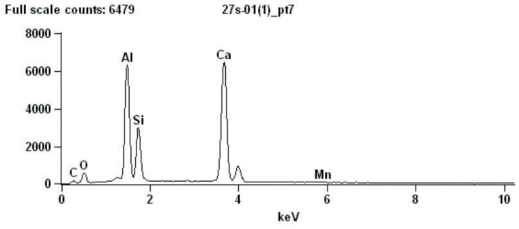

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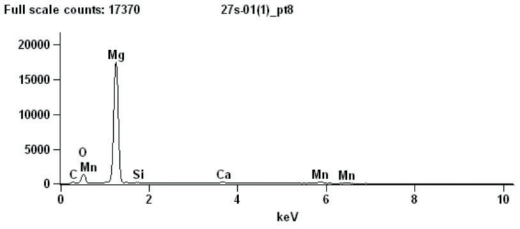

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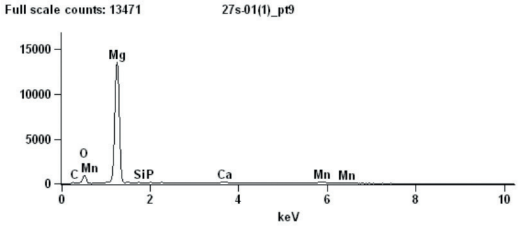

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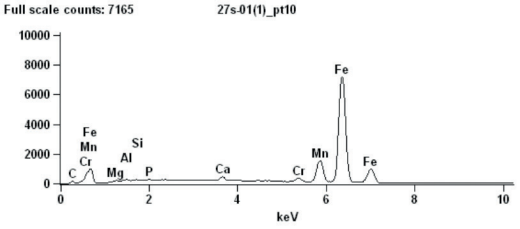

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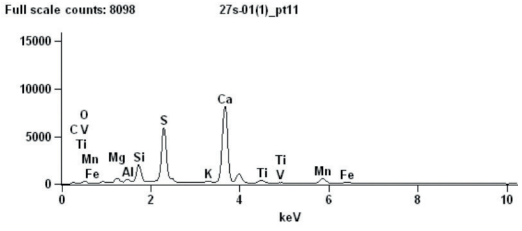


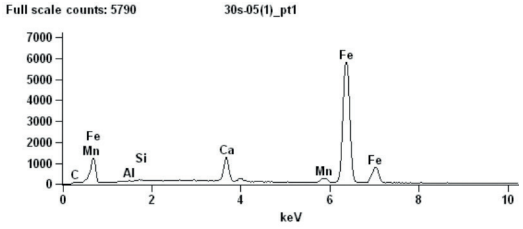
Table 4: *Chemical composition of slag phases according to Figure 6*

Point of analysis	Oxides [w/%]									Σ
	SiO_2	TiO_2	Al_2O_3	FeO	MnO	MgO	CaO	Cr_2O_3	P_2O_5	
1	0.35	-	0.19	61.69	19.58	-	18.19	-	-	100.00
2	0.70	1.69	5.60	9.15	2.90	-	79.96	-	-	100.00
3	-	-	-	22.46	64.89	5.80	2.51	4.34	-	100.00
4	19.64	-	-	-	-	-	78.66	-	1.70	100.00

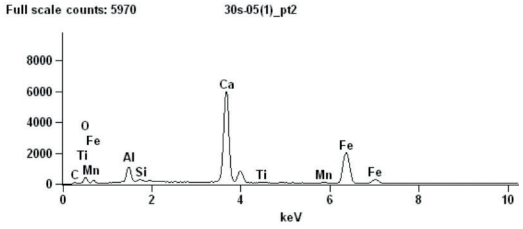
Explanations:
Points: 1 – solid solution of FeO-MnO-CaO, 2 – calcium oxide, 3 – solid solution of FeO-MnO, 4 – dicalcium silicate

Table 5: *EDS spectrums (Scanning Microscopy) according to Figure 6*

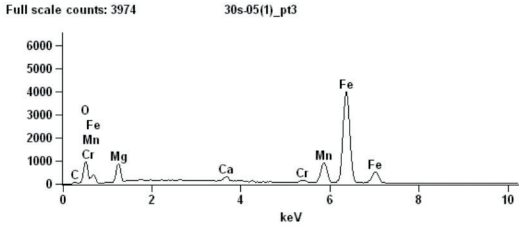
Point 1



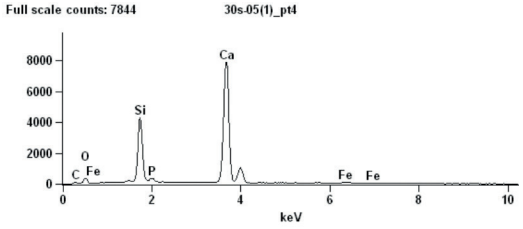
Point 2



Point 3



Point 4



Conclusions

Studied slags after steel production are characterized by diverse mineral and chemical composition. Among their components there can be distinguished: glaze, which is usually a dominant compound, metallic precipitations and non-metallic phases – oxides (mainly solid solutions of FeO, MnO, MgO and CaO) and silicates, represented by a large group of calcium silicates. In internal structures of silicates phases the presence of different elements substitutions, which are not present in nature, have been observed. Silicates which do not contain any substitutions are rare. Similar components may also be distinguished in slags from other steelmaking processes, but their quantitative participation and the content of admixtures in them are usually different. That is why, an individual approach to each type of studied slags is very important.

Metals may occur in metallurgical slags as fine drops not separated from slag during a metallurgical process, may form polymetallic aggregates, inclusions and its own phases (especially oxide ones), they can also hide in structures of silicate phases. Metals are also dispersed in glaze and amorphous substance.

It is very important to do mineralogical and chemical researches of slags to get to know what forms of metals occurrence there are, what the minerals resistance to weathering processes is and in which conditions metals are liberated from their components. This knowledge will be useful in economic activities connected with using metallurgical slag as a secondary material.

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Professional paper

Received: June 5, 2013
Accepted: November 20, 2013

Hydro-geophysical evaluation of groundwater potential in hard rock terrain of southwestern Nigeria

Hidrološko-geofizikalna opredelitev potenciala podtalnice v ozemlju trdnih kamnin v jugozahodni Nigeriji

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Abstract

In an attempt to characterize groundwater potential at the recently acquired land for University of Ibadan Cooperative Housing Estate located at Alabata near Ibadan, South-western Nigeria, integrated geophysical survey involving Very Low Frequency-Electromagnetic (VLF-EM) and resistivity methods were adopted. The VLF data measured along eight profiles were processed applying Fraser filtering and Karous-Hjelt filter on measured real components of the field data. Structural features significant to groundwater development were evident in the Fraser filter map and equivalent current density pseudo-sections. Thirteen Vertical Electrical soundings (VES) were carried out across the area using the Schlumberger electrode array configuration, with half-current electrode separation (AB/2) varying from 1 m to 100 m. The layer model interpretation obtained from the sounding curves revealed three to four layer earth models categorized into topsoil, lateritic hardpan, partially weathered layer and the fresh bedrock. The overburden thickness varies from 4.9 m to 19.1 m. Maps of the aquifer resistivity, aquifer thickness, overburden thickness, basement relief, bedrock resistivity, and secondary geoelectric (Dar-Zarrouk) parameters revealed delineated area with prolific aquiferous groundwater potentials.

Key words: Alabata area, geophysical investigation, current density map, geo-electric map, prolific zones.

Izvleček

Pri poskusu opredelitve potenciala podtalnice v zemljišču, nedavno nabavljenem za zadružno stanovanjsko gradnjo pri Ibadanski univerzi v Alabati pri Ibadanu v jugozahodni Nigeriji, so izvedli integrirano geofizikalno raziskavo, pri kateri so uporabili zelo nizkofrekvenčno elektromagnetno (Very Low Frequency-Electromagnetic -VLF-EM) in upornostno metodo. Podatke VLF-merjenj v osmih profilih so obdelali z uporabo Fraserjevega filtriranja in Karous-Hjeltovega filtra na merjenih realnih komponentah terenskih podatkov. Strukturne značilnosti, povezane s prisotnostjo podtalnice, so se pokazale na kartah Fraserjevega filtriranja in psevdopreseki ekvivalentne tokovne gostote. Na preiskovanem območju so izvedli trinajst vertikalnih električnih sondiranj (VES) po Schlumbergerovem razporedu elektrod s polovičnim razmikom tokovnih elektrod (AB/2) od 1 m do 100 m. Z interpretacijo plastovnega modela, dobljenega iz krivulj sondiranja, so postavili model treh do štirih plasti, ki ustrezajo tlom, trdni lateritni plasti, plasti delne preperine in neprepereli matični kamnini. Debelina krovnih plasti je od 4,9 m do 19,1 m. Iz kart upornosti v vodonosniku, njegove debeline, debeline krovnih plasti, reliefa podlage, upornosti podlage in sekundarnih geoelektričnih lastnosti (Dar-Zarrouk) je bilo mogoče določiti območja obetavne izdatnosti podtalnice.

Ključne besede: območje Alabata, geofizikalna raziskava, karta tokovne gostote, geoelektrična karta, cone izdatnosti

Introduction

University of Ibadan cooperative recently acquired a parcel of land to serve as housing estate for its members. The basement complex rocks of southwestern Nigeria underlie the estate, which is located at Alabata near Ibadan. In typical hard rock areas, the geological sequence normally encountered is characterized by the existence of basement rock overlain by variable unconsolidated materials referred to as overburden. The groundwater in a typical Basement Complex environment is usually contained in the weathered and/or fractured basement rocks or alluvial deposits within flood plains.[1] However, the discontinuous nature of the basement aquifer system makes detailed knowledge of the subsurface geology, its weathering depth and structural disposition through geologic and geophysical investigations inevitable.[2] In order to evolve a pragmatic and scientific planning for the management of groundwater resources in this estate, a hydro-geophysical evaluation of the groundwater potential was carefully carried out.

Integrated geophysical tools, especially resistivity and electromagnetic methods, are commonly used in groundwater exploration, mainly due to the close relationship between electrical conductivity and some hydrological parameters. The Very Low Frequency Electromagnetic (VLF-EM) is an effective tool in mapping conductive fault and fracture zones while resistivity method is used for detecting groundwater presence and differentiating subsurface layers. Electrical and electromagnetic geophysical methods have been widely used in groundwater investigations because of good correlation between electrical properties, geological (composition) and fluid content.[3–6] In present paper, Very Low Frequency (VLF-EM) and Vertical Electrical Sounding (VES) methods were employed to evaluate the groundwater potential of University of Ibadan cooperative housing estate in Alabata, Ibadan.

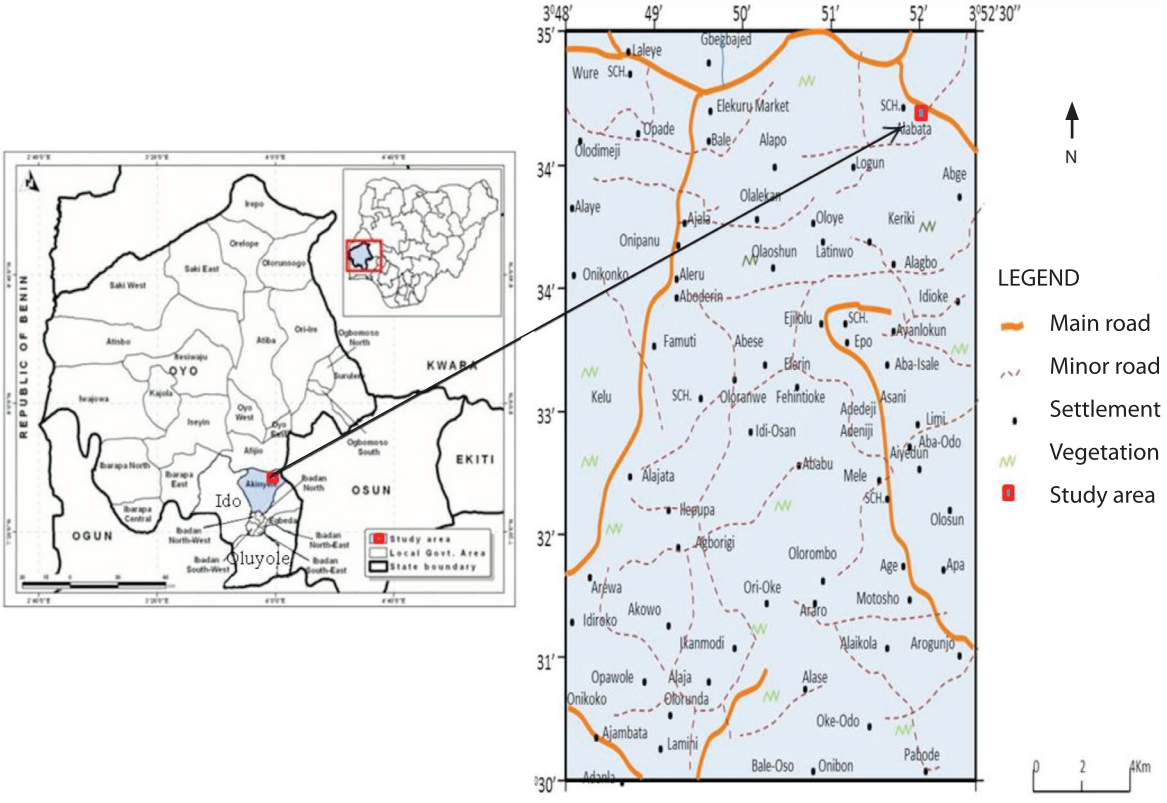

Figure 1: *Location map of the study area.*

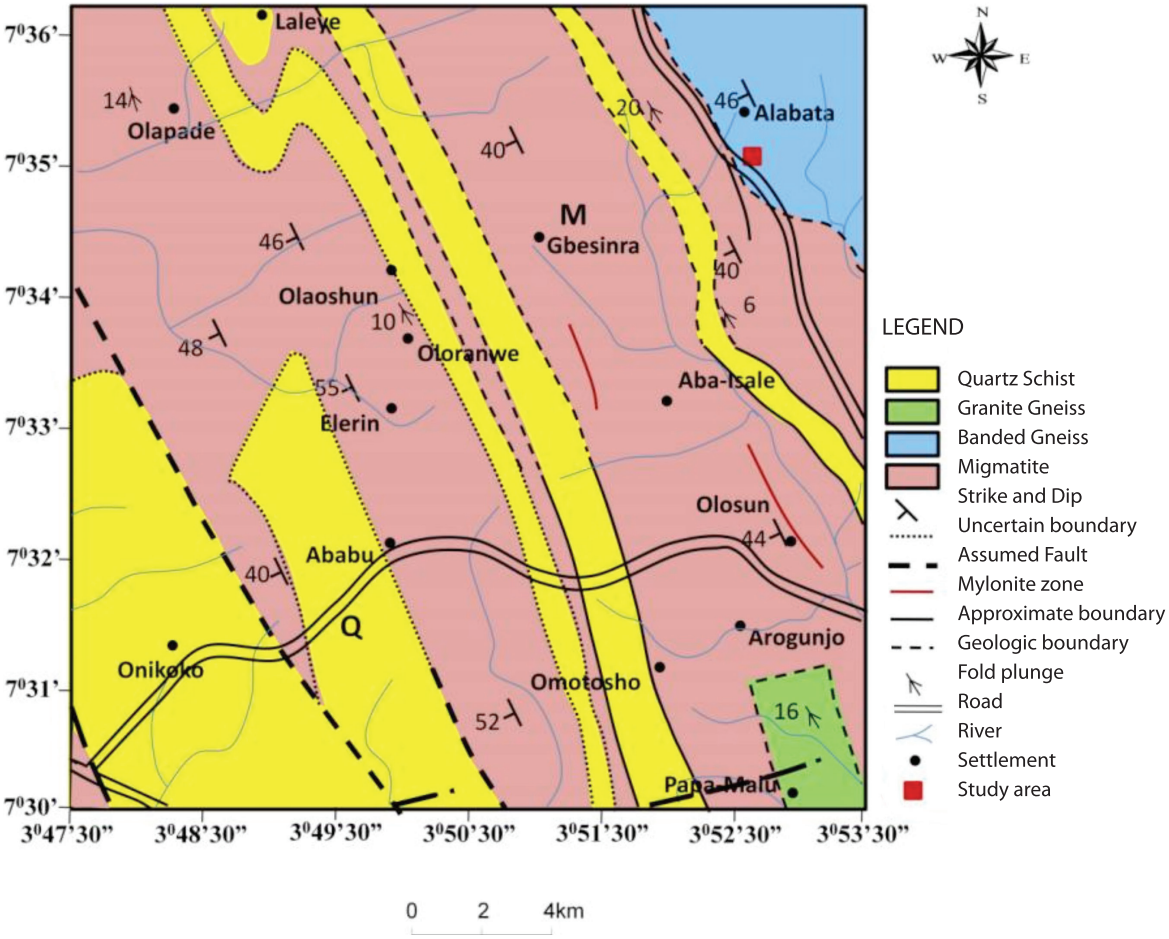

Figure 2: *Geological map of the study area (Afenkhare, 2012).*

Site Description and Geological Setting

The study area is located in Alabata, Ibadan, southwestern Nigeria (Figure 1). It is confined within latitudes 7° 34.970 and 7° 35.138 and longitudes 3° 52.180 and 3° 52.0. The study area is characterized by relatively gentle undulating terrain with elevations of between 265 m and 278 m above mean sea level (msl). The vegetation in the area is of rainforest type, characterized by short dry season and long wet season, with high annual rainfall ranging between 1 000 mm and 1 200 mm. Annual mean temperature is between 22 °C and 33 °C with relatively high humidity.[7] The survey area is underlain by the Precambrian basement complex rock of southwestern Nigeria. Metamorphic basement rocks, mostly undifferentiated migmatite-gneiss, quartzite-schist, banded gneiss and granite gneiss, underlie the area.[8] Figure 2 highlights the local geology of study area. The study area falls in the area underlain by banded gneiss in Alabata. The coarse-grained banded gneiss was low-lying with the elevation ranging from 240 m to 290 m (msl). It strikes approximately north-south with minor folds. There are quartz and pegmatite intrusions occurring concordantly with the rock's strike direction.

Materials and methods

The field investigation involved application of both Very Low Frequency Electromagnetic (VLF-EM) measurements and Vertical Electrical Sounding (VES) for mapping fractures in the bedrock and delineating geoelectrical layers in the overburden materials.

VLF measurement

VLF surveying falls into the far-field system of electromagnetic data collection. The VLF transmitter is a military-based communications antenna that emits a very powerful electro-

magnetic wave, which when detected tens of kilometers from the source, behaves as a horizontally propagated plane wave.[9] The propagating signal has horizontal and linearly polarized magnetic and electrical components of the radio-wave field in the absence of a subsurface conductor. However, eddy currents are generated when the radio-wave field passes through a buried conductor, creating a secondary electromagnetic field. The increase in the flow of induced current causes the magnetic field to tilt in the vicinity of conducting structures.[10] Since this causes a phase shift with respect to the homogeneous primary field, the total field is elliptically polarized and tilts with respect to the horizontal axis. Consequently, tilt-angle variations follow a response across the anomaly and thus the crossover point coincides with the center of the anomaly.

Many commercial instruments measure the changes in the different parameters of the total field. For example, some instruments measure the dip of the major axis and the ellipticity of the polarization ellipse; whereas other instruments measure the vertical and horizontal field components. These components of the anomalous field can be converted into ratios of the vertical anomalous field to the horizontal primary field for tilt angle analysis. Further, a current density can be calculated with respect to depth from the measured magnetic field. For example, a buried sheet conductor in a resistive medium in a horizontal primary magnetic field will induce changes in the amplitude and direction of the primary field in proximity to the target. Consequently, on one side of the target, the angle between the vectors of the primary and secondary components of the radio wave field will reach a maximum near an object and change to a minimum upon passing a buried target. The point at which the tilt angle passes through zero, the "crossover" point lies immediately above the target.[11] If the target dips, then the tilt-angle measurements on one side of the anomaly are accentuated at the expense of the tilt-angle measurements on the other side of the target. The tilt angle and current density derived from the anomalous magnetic field can be used in subsequent statistical analyses to locate and to image the subsurface target.

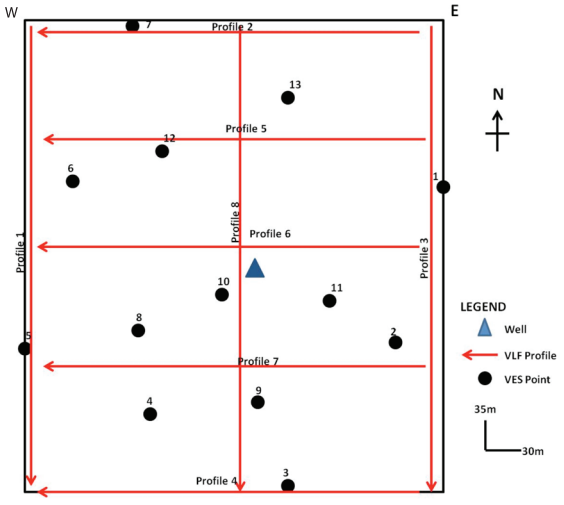

Figure 3: *Location map of the study area showing the VLF-EM profiles, VES points and dug well.*

Linear filtering of the tilt-angle measurements can aid in locating the position of a buried target. Fraser[12] proposed a simple linear statistical filter of tilt-angle data that converts tilt-angle crossovers into peaks for ease of analysis. Fraser filtering consists of averaging the tilt-angle measurement produced by a subsurface conductor. In a linear sequence of tilt-angle data M_1, M_2, M_3 ... M_n measured at a regular interval, the Fraser filter F_i is:

$$\Phi_1 = (M_3 + M_4) - (M_1 - M_2) \quad (1)$$

The first value F_1 is plotted half way between positions M_2 and M_3; the second value is plotted halfway between M_3 and M_4.

Many instruments can calculate a current density from the magnitude of the measured magnetic field.[13] Karous and Hjelt[14] developed a statistical linear filter, based upon[12] and linear field theory of Bendat and Piersol[15] This filter provides an apparent depth profile from the current density (H0) which is derived from the magnitude of the vertical component of the magnetic field at a specific location (Figure 3). The depth profile can be calculated from:

$$I_a(0) = 2\pi\,(-0.102H_{-1} + 0.059H_{-2} - 0.561H_{-1} + \\ + 0.561H_1 - 0.059H_2 + 0.102H_3)/Z \quad (2)$$

Where, the equivalent current density I_a at a specified horizontal position and depth Z is based upon a symmetrical filter of the measured current (from the measured magnetic component of the anomalous field).

In this study, VLF-EM method was employed to map the study area with the object of isolating fracture zones which are likely to be filled with water. ABEM Wadi VLF electromagnetic equipment with in-built digital display unit and powered by battery was used. For the VLF-EM measurements, radio signal from station GQD in Rugby UK was the main signal station tuned / selected. This corresponds to frequency values of 18.8 kHz and was employed to generate the primary electromagnetic field around the buried conductors in order to induce the detected secondary field and measured as a fraction of the primary field by the VLF-meter.

Eight profiles were measured with three (3) trending approximately N–S and five (5) trending approximately E–W with measurement station intervals of 10 m. The profiles ranges between 170 m and 250 m long and the majority of the profiles run perpendicular to the general N–S geologic strike in the study area (Figure 3). A sub-meter-accurate Global Positioning System (GPS) was used for exact spatial positioning of collected data.

Geoelectric resistivity measurement

Electrical resistivity data were acquired using the Campus Ohmega resistivity meter. The survey involved 1-D Vertical Electrical Sounding (VES). The VES utilized the Schlumberger electrode array with half-current electrode separation (AB/2) ranging from 1 m to 100 m and thirteen (13) VES stations were occupied (Figure 3). The coordinates of each VES station were taken with the Garmin handheld Global Positioning System (GPS) device to ensure accurate future geo – referencing

Data Processing and Evaluation

The VLF-EM data as well as those of the VES measurements were subjected to data processing and evaluation as the basis for interpretation.

For VLF-EM, the acquired field data were processed to simplify the obtained complex information into a profile in which the displayed function is directly related to physical property of the underlying rock. Thus, measured raw real and imaginary components were subjected to Fraser[12] and Karous-Hjelt[14] filtering operations to suppress noise and enhance signal. The Fraser filter[12] converts crossover points into peak responses by 90° phase shifting. This process removes direct current bias that reduces the random noise between consecutive stations resulting from very low frequency component of sharp irregular responses.[16] The Karous-Hjelt filter[14] uses the linear fit theory to solve the integral equation for the current density. This forms the basis of the overall interpretation and delineation of potential fracture zone.

The VES, field data were interpreted through the following steps:

- smoothing of the apparent resistivity field data curve and removing the electrical noises superimposed using an appropriate filter operator;[17]
- matching the smoothed field curve with the standard curves of the auxiliary method;[18, 19]
- preparing an initial geo-electrical model (thicknesses and corresponding resistivities) for a limited number of layers and incorporating the geological background and well information in the study area;[6]
- entering the initial geo-electrical model into the Vander Velpen[20] modeling package. Iterations were carried out to reach the best fit between the smoothed field curve and the calculated one. The root mean square (RMS) errors of the resulting models ranged between 2.3 % and 3.2 %. The final VES interpretation results (layer resistivities and thicknesses) were used to generate secondary geoelectric (Dar-Zarrouk) parameters, weathered layer thickness map, weathered layer resistivity map, overburden thickness map, bedrock resistivity map and the basement topography map of University of Ibadan cooperative housing estate in Alabata, Ibadan. The spatial representation of the data was done using surfer 9.0 software with Kriging employed as the gridding method. The data were ranked using the overburden thickness, aquifer resistivity, aquifer thickness and bedrock topography inferred from the first order geoelectric parameters and total longitudinal unit conductance, total transverse resistance unit and electrical anisotropy inferred from second order geoelectric parameter to generate the groundwater potential map of the study area.

Geoelectric (Dar-Zarrouk) Parameters

A geo-electric layer is described by two fundamental parameters: its resistivity (ρ_i) and thickness (h_i), where the subscript i indicates the position of the layer in the section. Other geoelectric parameters can be derived from its resistivity and thickness.[21] For $i = 1, 2 \ldots n$-layer, these parameters are:

— Total longitudinal conductance (S)
$S/\Omega = h_1/\rho_1 + h_2/\rho_2 + \ldots + h_n/\rho_n$

Total transverse resistance (T)
$T/(\Omega\ m^2) = h_1\,\rho_1 + h_2\,\rho_2 + \ldots + h_n\,\rho_n$

Maillet[22] has defined S and T as Dar-Zarrouk parameters. They can be defined for individual layers, or as a summation for a multi-layer section.

— Average longitudinal resistivity (ρ_L)
$\rho_L/(\Omega\ m) = H/S = \Sigma\, h_i/(\Sigma h_i/\rho_i)$

Where $H = \Sigma\, h_i$ (h_i is the thickness for each layer i)

— Average transverse resistivity (ρ_t)
$\rho_t/(\Omega\ m) = T/H = (\Sigma h_i\,\rho_i)/\Sigma h_i$

— Electric anisotropy (λ)
$\lambda = (\rho_t/\rho_L)^{1/2} = (T\,S/H^2)^{1/2}$ (dimensionless)

— Root means square resistivity (ρ_m)
$\rho_m/(\Omega\ m) = (\rho_t \times \rho_L)^{1/2} = \lambda \times \rho_L = (1/\lambda) \times \rho_t$

In this study area, the above geoelectric parameters (S, T, ρ_L and ρ_m) are calculated to the top of the basement rock as shown in Table 1.

Table 1: *Calculated geoelectric (Dar-Zarrouk) parameters*

VES NO	LATITUDE*	LONGITUDE*	Elevation (m)	ℓ_1 (Ω m)	ℓ_2 (Ω m)	ℓ_3 (Ω m)	ℓ_4 (Ω m)	h_1 (m)	h_2 (m)	h_3 (m)	S (1/Ω)	T (Ω m²)	λ	M (m)
1	596129.48	838398.80	278.5	113.8	115.8	1823.6	_	1.7	5.6	_	0.063 3	841.94	1.00	267.3
2	596107.62	838297.41	277.1	221.8	57.2	3643.6	_	1.1	7.3	_	0.132 6	661.54	1.11	259.6
3	596058.17	838199.66	270.6	294.8	740.9	620.5	2 489.9	1.2	7.1	10.8	0.031 1	12 315.55	1.02	248.9
4	595993.71	838252.97	268.3	326.8	53.5	5365.4	_	0.8	4.1	_	0.079 1	480.79	1.26	268.1
5	595936.63	838293.39	264.9	110.6	72.8	1155.4	_	1.1	7.0	_	0.106 1	631.26	1.01	257.9
6	595958.47	838402.14	270.4	440.0	35.4	1146.0	_	2.2	8.3	_	0.239 5	1 261.82	1.66	256.5
7	595987.68	838509.07	272.0	58.0	158.8	37.9	1 906.6	1.8	5.1	11.3	0.361	1 342.55	1.00	247.8
8	595989.92	838509.07	268.3	427.0	46.6	1230.4	_	1.2	6.2	_	0.135 9	801.32	1.44	276.6
9	596045.18	838308.23	269.9	357.9	41.9	739.6	_	1.2	7.0	_	0.170 4	722.78	1.35	263.8
10	596026.65	838260.44	273.2	702.3	51.5	2803.9	_	1.0	6.2	_	0.121 8	1 021.6	1.01	269.8
11	596076.30	838328.58	271.2	153.4	29.0	1813.3	_	1.7	6.1	_	0.221 4	437.68	1.26	267.2
12	595998.88	838422.49	274.8	237.4	57.3	792.4	_	2.0	11.5	_	0.209 1	1 133.75	1.14	264.5
13	596055.81	838457.61	274.0	127.6	148.1	133.3	2 709.9	1.5	10.7	4.2	0.115 5	2 335.93	1.00	263.6

*Date for geographic coordinates is Universal Transverse Mercator (UTM)
$\ell_1 - \ell_4$ = Resistivity values for each layer (Ω m)
$h_1 - h_3$ = True thickness for each layer (m)
S = Total longitudinal conductance (1/Ω) to the top of the basement rock
T = Total transverse resistance (Ω m²)
λ = Electric anisotropy (dimensionless) to the top of the bedrock
M = Bedrock relief (m)

Results and discussion

VLF-EM Survey

Fraser filtering responses ranged in value from –105 % to 160 % along the profiles. Figure 4 shows the Fraser filtered data (real or in-phase components). The in-phase profiles show positive peaks of different intensities and sharpness, suggesting the presence of shallow and deep conductors.[23]

Lower values of relative current density correspond to higher values of resistivity.[24] All the VLF-EM profiles in this study were processed using the Karous–Hjelt filter.[25] Conductors (coloured red) were delineated from equivalent current density pseudo sections along

traverse 1, 3 and 7 (Figure 4). A higher value of relative current density is regarded as conductive subsurface structures, such as fractures,[23, 26] which often store groundwater in hard rock terrains.

The 2-D inversion shows the variation of equivalent current density, and change in conductivity with depth. With such equivalent current density cross-section plots, it is possible to qualitatively discriminate between conductive and resistive structures where a high positive value corresponds to conductive subsurface structure and low negative values are related to resistive materials.[24, 26] In addition, equivalent current density cross-section also gives an idea about the dip direction; however, exact dip angle cannot be estimated due to the vertical axis variable being a pseudo depth only.[26, 27]

The equivalence current density pseudo-section of profile 1 (Figure 4a) reveals the presence of major anomaly at the southern section between 125 m and 162 m, which can be referred to as fracture zone.[28] Furthermore, two high current density zones between 17 m and 26 m, and 75 m along the profile can also be referred to as indications of the potential subsurface fracture system[26] with the fracture at 75 m dipping southwest (Figure 4a). Asymmetry in the observed real and imaginary anomalies suggests the dipping nature of a subsurface conductive body.[29, 30] The Fraser filtering data plots and the Karous-Hiljet current density plot for profile 3 as presented in Figure 4a reveals a number of anomalies, which reflects conductive subsurface structural trends of inferred fractures zones. In addition, profile 7 shows

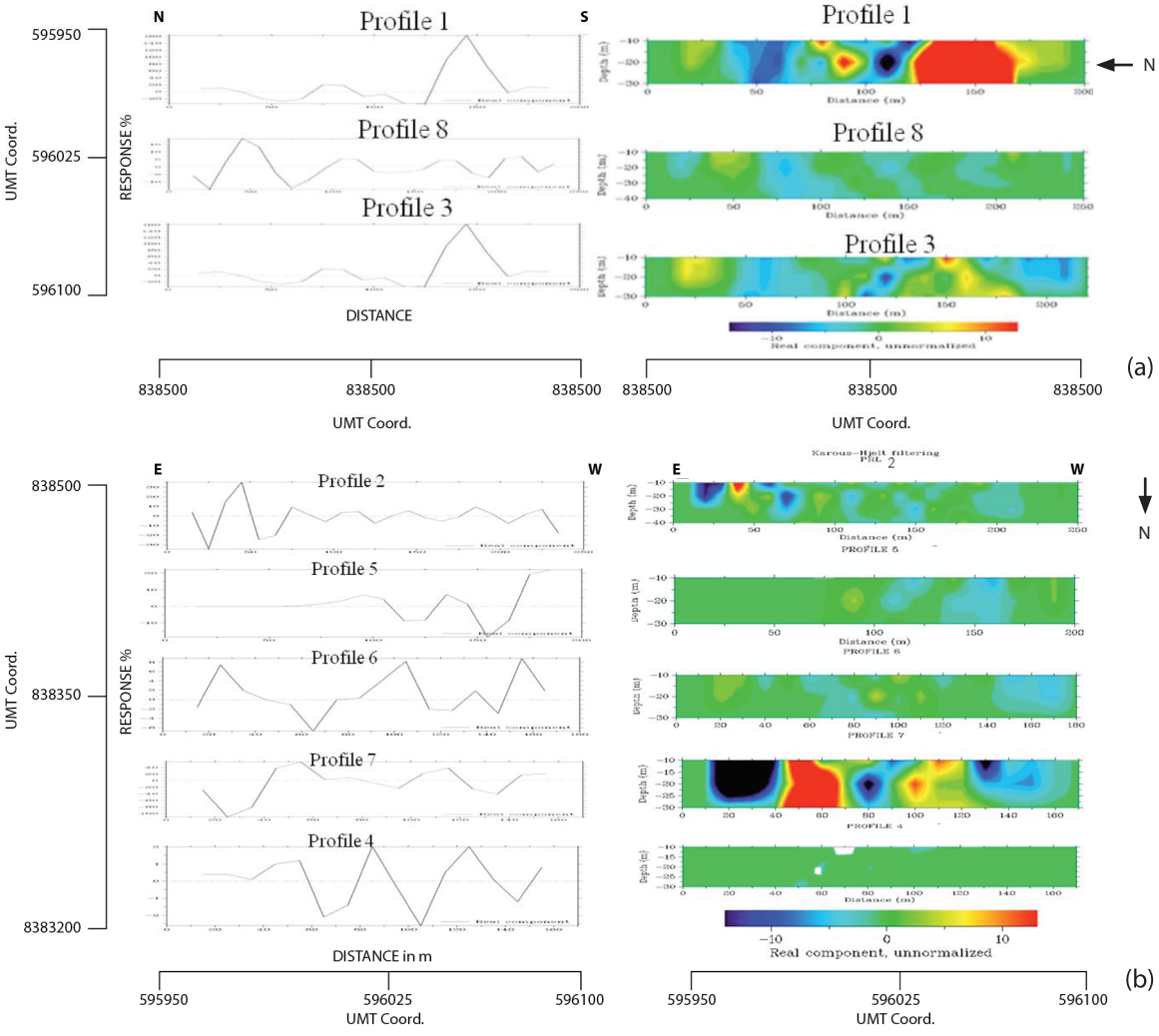

Figure 4: *Fraser filtering graph and equivalent current density pseudo-sections: (a): N-S direction, (b): E-W direction.*

high equivalent current density between stations 42 m and 68 m and station 110 m with the latter dipping southeast. Other closures of conductive bodies are present on different section with each conductive body coinciding with points identified on the profiles as fractures and or geological features (Figures 4a and b).

Resistivity Sounding Curves

The resistivity sounding curves obtained from the study area varied from the 3-layer (A and H types) to 4 layer (KH) with the H type being the predominant. The typical curve types are as shown in Figure 5. Table 1 gives the summary of the VES interpretation. The thickness and characteristics of the aquifer are fairly known due to the well dug in the centre of the study area. The key to success of any geophysical survey is the calibration of the geophysical data with hydro-geological and geological ground truth information; therefore, geoelectric station 10 was purposely located near the well (Figure 3). Measurements from existing well dug in the area reveal lateritic top soil, sandy clay, and basement rock. The depth of the well measured is 6.2 m and the potentiometric surface is at 5.3 m. The depth measurement correlates fairly well with the interpreted values of VES-10 (Figure 5).

Weathered Basement (Aquifer unit) Resistivity Map

The weathered basement resistivity map (Figure 6) shows the resistivity variation within the aquifer units of the study area. The resistivity value range is between 29 Ω m and 621 Ω m; with a mean value of 104 Ω m. The resistivity is least at the centre towards the western part of the study area with values ranging between 29 Ω m and 100 Ω m. The northern part has resistivity values ranging from 100 Ω m to 150 Ω m with the resistivity increasing towards the north. The resistivity also increases toward the south of the study area with the values ranging between 100 Ω m and 620 Ω m (Figure 6). The classification of the groundwater potential of the aquifer units based on resistivity was premised on the findings of Adiat et al.[2] Zones characterized by resistivity value less than 100 Ω m or greater than 400 Ω m was recognized as area of least groundwater prospect. Zones of medium yield

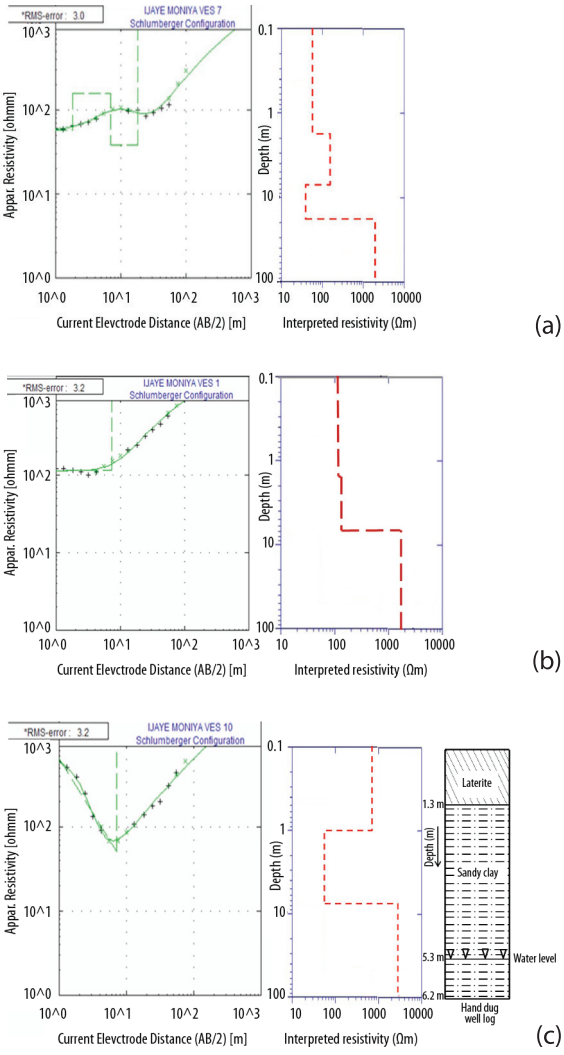

Figure 5: *Representative VES curve in Alabata with their respective interpreted resistivity log. (a): KH curve, (b): A curve & (c): H curve.*

for groundwater prospect are characterized by resistivity values ranging between 300 Ω m to 350 Ω m. Areas with resistivity values between 100 Ω m and 300 Ω m are classified as zones of high groundwater yield potential. In addition, Olayinka et al.[5] classified weathered basement in the basement complex of Nigeria with resistivity values ranging from 100 Ω m to 800 Ω m as good groundwater aquifer. Barker et al.[31] also observed that the highest yielding boreholes in the basement complex of Zimbabwe were associated with weathered layers resistivity values between 100 Ω m and 600 Ω m. Based on these, the northern and southern part of the study area is presumed to have good groundwater aquifer.

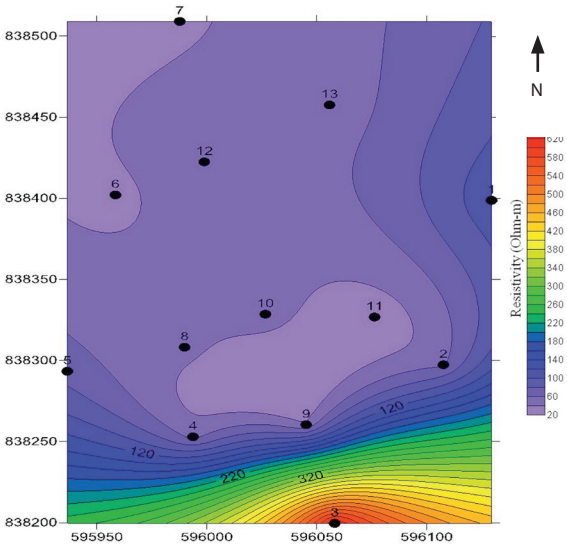

Figure 6: *Aquifer resistivity map of the study area.*

Weathered Basement (Aquifer Unit) Thickness Map

The thickness of the weathered basement varies between 4.1 m and 11.5 m (Table 1). The aquifer unit in the study area has a mean thickness of 7.4 m. The aquiferous zone is relatively thick around the northwest and southeast portion of the study area (8 m to 11.5 m) while the thickness of the remaining part is relatively thin (4.1 m to 8 m) (Figure 7). The aquifer unit in the entire area is generally characterized by low thickness between 4 m to 8 m. However, some areas have relatively thick aquiferous unit with thickness varying between 8 m and 12 m.

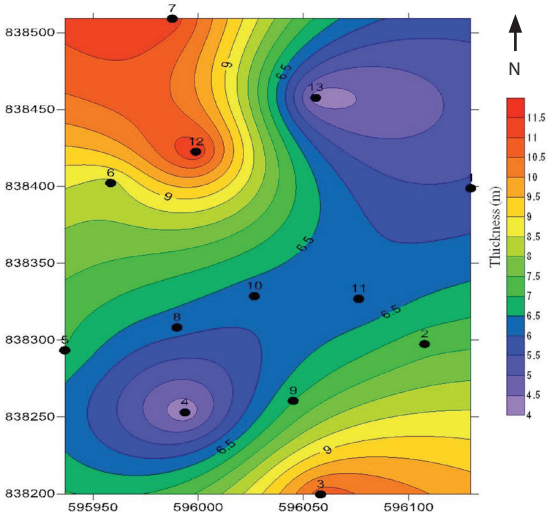

Figure 7: *Aquifer thickness map of the study area.*

Overburden Thickness Map

The overburden thickness map (Figure 8) shows that the overburden thickness of the area varies from 4.9–19.1 m, with a mean of 10.5 m. The overburden thickness map show zones of relatively thick overburden (greater than 13 m) and zone of relatively thin overburden (< 13 m). Appreciable overburden thickness zones are possible groundwater collecting zones; therefore, unconsolidated material could contain reliable aquifer if thick and sandy.[5, 32] Geophysical studies in southwestern basement complex of Nigeria have identified thick overburden as zones of high groundwater potentials.[33–35] The overburden is relatively thick (13 m to 20 m) in the northern and southeastern portions of the study area. These zones are suggestive of possible groundwater potential zones in the area. Such zones cover about 38 % of the entire study area.

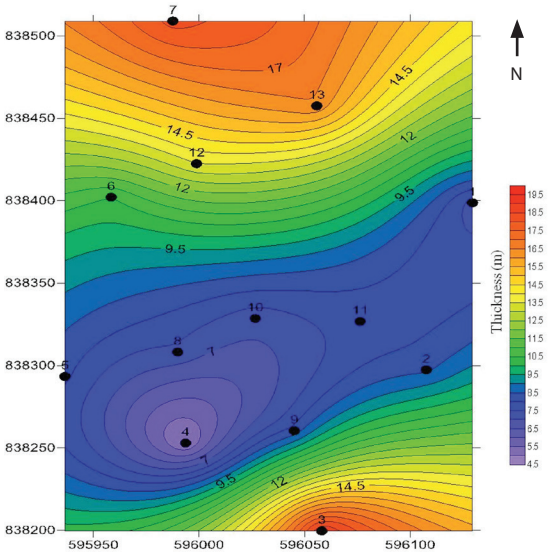

Figure 8: *Overburden thickness map of the study area.*

Aquifer protective capacity evaluation

The total longitudinal conductance (S) to the top of the basement rock ranges between 0.031 1 S and 0.361 S. The maximum value was recorded at VES 7 (0.361 S) with gradual decrease towards the south (Figure 9). The minimum value was recorded at VES 3 (0.031 1 S). A marked increase in *S* may correspond to an average increase in the clay content and consequently a decrease in transmissivity.[6] The total longitudinal unit conductance values can also be utilized in evaluating overburden protective capacity in an

area.[36] This is because the earth medium acts as a natural filter to percolating fluid. Its ability to retard and filter percolating fluid is a measure of its protective capacity.[36, 37] The highly impervious clayey overburden, which is characterized by relatively high conductance, offers protection to the underlying aquifer.[38] The protective capacity of the overburden has been zoned into good, moderate and weak protective capacity.[39] They classified the longitudinal conductance above 0.7 S as good protective capacity zone, the portion having conductance values ranging from 0.2 S to 0.69 S were classified as zone of moderate protective capacity while zone with 0.1 S to 0.19 S was classified as weak protective capacity and where the conductance value is less than 0.1 S were considered poor. The above classification has revealed that the overburden materials of the study area ranging between moderate to poor protective capacity zone. The moderate protective capacity covers the northwestern part of the study area and extends towards the central part, which has weak protective capacity. The northeast through the eastern part to the south of the study area have poor protective overburden (Figure 9). From the longitudinal conductance map of the area, about 30 % of the area falls within the moderate protective capacity while about 70 % constitutes the weak/poor protective capacity rating. This suggests that materials of weak/poor protective capacity underlie the area.

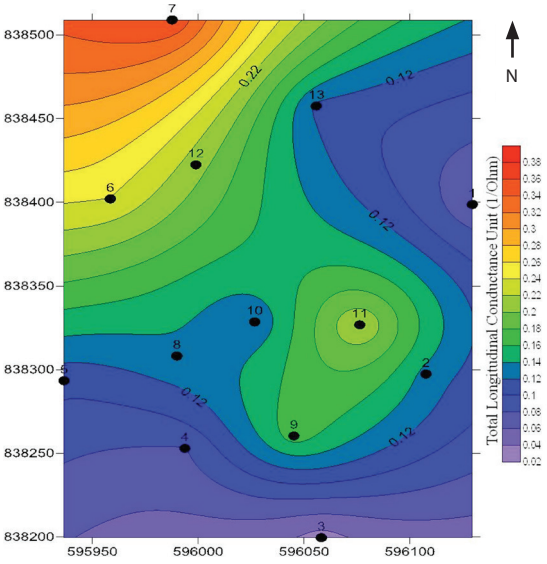

Figure 9: *Total longitudinal conductance unit map of the study area.*

Bedrock Topography Map

The bedrock topography map (Figure 10) reflects the topography of the bedrock underlying the area and its structural disposition. The map shows that the basement structures in the area include both basement ridge and depressions. The ridge occupies the eastern and central parts of the study area while the depressions occupy the northern, western and southern parts of the study area. Naturally, the groundwater flows from areas of high pressure (such as bedrock ridge) to area of low pressure (such as bedrock depression). It is then expected that areas identified as depressions on the map are the groundwater collection points, which have significant role in groundwater development.

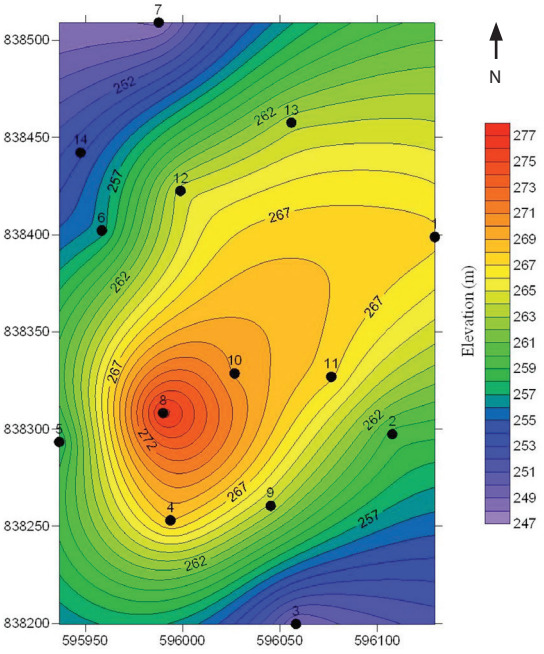

Figure 10: *Bedrock topography map of the study area.*

Electrical anisotropy (λ)

In the study area, the coefficient of anisotropy to the top of the basement rock ranges between 1.00 and 1.66. The maximum λ value was recorded at VES-6 (1.66) and the minimum λ values are recorded at VES-1, 7, and 13 (1.00) (Table 1). The coefficient of anisotropy map (Figure 11) shows that λ is high around the western part of the study area and decreases towards other zones. Singh and Singh[40] pointed out that lower values of anisotropy correspond to high aquifer potential zones. Based on this, the northern portion through the east to

the southern part of the study area is characterized by higher groundwater potential. These areas coincide with the areas with weak/poor protective layers (Figure 9); part of it falls on the ridge in the bedrock topography map (Figure 10), which is the groundwater-diverting zone. This is confirmed by the Fraser filter and equivalent current density distribution in profile 4 (Figure 4b) with high response on Fraser filter graph but with no corresponding accumulation of current density distribution on the equivalent current density pseudo-section. This fracture may contain unsaturated material. However, the depression indicated on the bedrock topography map (Figure 10) contains saturated material presumed to be groundwater as indicated on Fraser filter and equivalent current density pseudo-section of profile 6 (Figure 4b). In addition, the southern part of this area has the requirements that favour groundwater abstraction (Figure 11).

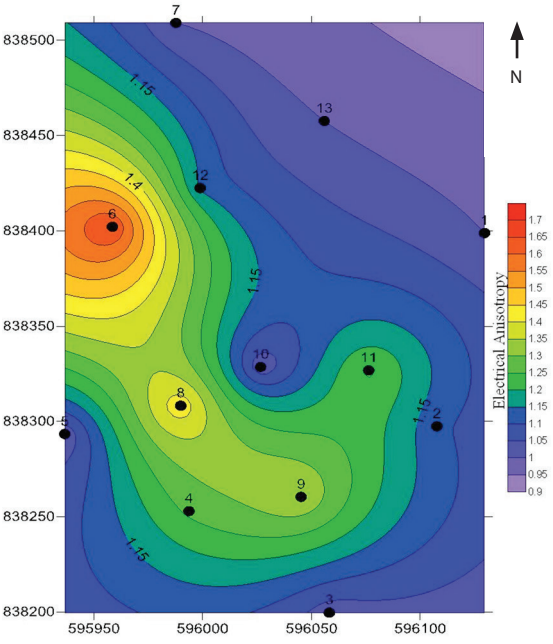

Figure 11: *Electrical Anisotropy map of the study area.*

Bedrock resistivity map

Figure 12 shows the contour map of the bedrock resistivity. The resistivity values of the bedrock vary from 740 Ω m to 5 365 Ω m. According to Olayinka and Olorunfemi,[41] the resistivity values that exceed 1 000 Ω m is fresh bedrock but where the resistivity reduces to less than 1 000 Ω m, the bedrock is fractured

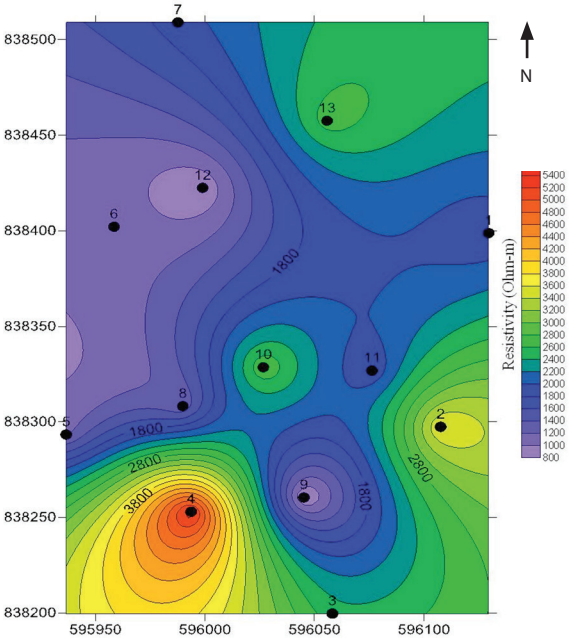

Figure 12: *Bedrock resistivity map of the study area.*

and saturated with fresh water. The fractured zone constitutes a major component of the aquifer in a basement complex area. From the map (Figure 12), the bedrock in the southwestern, southeastern, northeastern and around the central parts is highly resistive (2 000 Ω m to 5 400 Ω m). This coincides with the low conductivity values displayed on the equivalent current density pseudo-section of profiles 1, 2, 5, 7 and 8, which confirms the presence of highly resistive features in those zones. The resistivity values for the bedrock in the northwest to the major parts of the central portion towards the south central ranges between 1 200 Ω m to 2 000 Ω m. There is good correlation between bedrock resistivity map and the equivalent current density pseudo-sections crossing this area, which show shallow conductive features. The resistivity value of the western and minute portion of south central is less than 1 000 Ω m (700 Ω m to 1 000 Ω m). This is revealed by the current density pseudo-sections of profile 1, which shows a highly conductive feature between stations 122 m and 167 m, and profile 7, which has high current density reflection at station 110 m with the fracture dipping southeast. The prominent current density reflects the presence of localized fractures containing groundwater.[26] There is good correlation between the equivalent current density pseudo-sections and the bedrock resistivity map.

Total transverse resistance unit

The total transverse resistance unit (*T*) to the top of the basement rock ranges between 12 315.55 and 437.68 Ω m^2. The maximum *T* value was recorded at VES-3(12 315.55 Ω m^2) with gradual decrease towards the central part. The minimum *T* value was recorded at VES-11 (437.68 Ω m^2) Figure 13 shows the map of total transverse conductance unit of the study area. The southern part has the highest value, which ranging from 1 000 Ω m^2 to 12 300 Ω m^2. The central part has value ranging between 400 Ω m^2 to 1 000 Ω m^2 while the northern zone has value ranges from 1 000 Ω m^2 to 2 500 Ω m^2. Transverse resistance unit map has been used in determination of zones with high groundwater potential.[42] According to Braga et al.[43] high values of *T* can be associated with the zones of high transmissivity and areas with high values on *T* map indicate unconfined aquifer. Hence, the southern zone is suitable for groundwater exploitation.

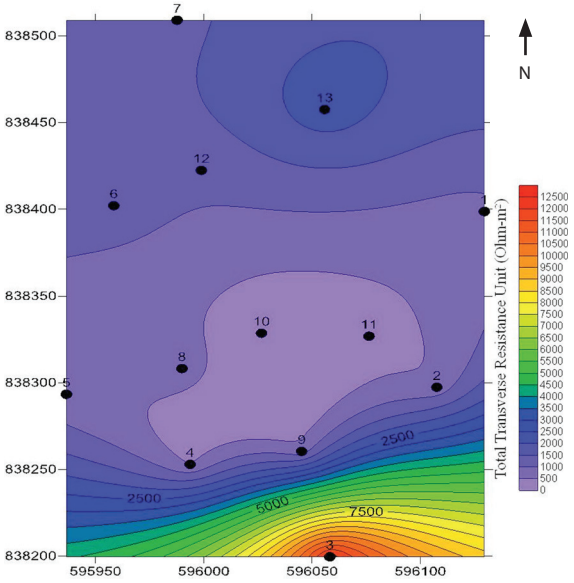

Figure 13: *Total transverse resistance unit map of the study area.*

Groundwater Potential Evaluation

The groundwater potential evaluation of the area was based on the integration of the equivalent current density pseudo-sections, aquifer resistivity, aquifer thickness, overburden thickness, total longitudinal conductance unit, total transverse resistance unit, and electric anisotropy and bedrock topography maps. The groundwater prolific area will have high accumulation of current density, which reflects the presence of fracture zones,[26] thick overburden > 10 m[5] and aquifer resistivity ranging from 100 Ω m to 800 Ω m.[2, 5, 31] In addition, groundwater potential area should have low longitudinal conductance unit, which indicate an increase in transmissivity,[6] high transverse resistance unit,[43] low values of electrical anisotropy < 1.2[40] and areas characterized by depressions on the basement topography map. These maps were synthesized and integrated for the evolvement of the groundwater potential map, which was eventually used to categorize the study area into good, moderate and poor groundwater potential zones (Figure 4). The central/western portion is characterised by decrease in overburden thickness (7.2 m at VES 10), weathered layer resistivity (35 Ω m at VES 6), total transverse resistance unit (723 Ω m^2 at VES 9) and increase in electric anisotropy (1.66 at VES 6) and total longitudinal conductance (0.24 S at VES 6), reflecting low aquifer potential. On the other hand, northeastern/southeastern region is characterized by increase in overburden thickness (19.1 m at VES-3), weathered layer resistivity (602 Ω m at VES-3), total transverse resistance unit (12 316 Ω m^2 at VES 3) and decrease in electrical anisotropy (1.00 at VES-1, 7 and 13) and total longitudinal conductance unit (0.031 1 S at VES-3), reflecting high aquifer potentials. In this regard, the northern and south-eastern parts of the study area are

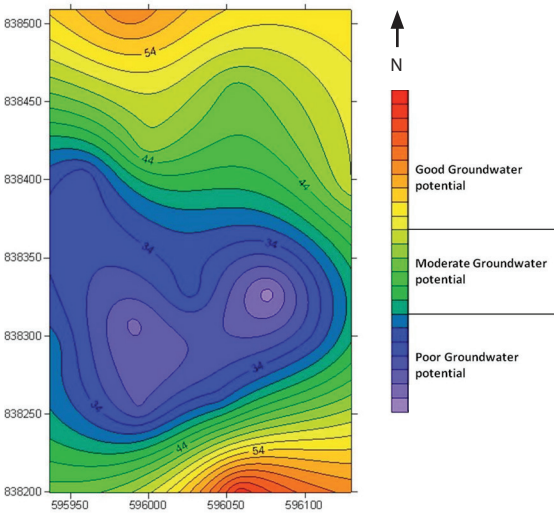

Figure 14: *Groundwater potential map of the study area.*

categorised as good groundwater potential; moving towards the central from the northern and southern parts, groundwater potentiality changes from good to moderate while the western/central part is categorised as area with poor groundwater potential (Figure 14).

Conclusions

A comparative integrated interpretation of VLF-EM and VES data enabled the evaluation of the groundwater prospect of University of Ibadan cooperative housing estate in Alabata, Ibadan; a basement complex terrain of south-western Nigeria. With the additional information obtained from existing well in the area, the spatial distribution of the regolith/weathered layer, containing the near-surface or overburden aquifers, was reliably delineated from the bedrock housing the bedrock aquifers. The geoelectric parameters (layer resistivities and thicknesses) which are known to be of hydrogeologic relevance, gathered from the VES interpretation were used to generate maps (weathered/fractured layer resistivity map, weathered/fractured layer thickness map, overburden thickness map, basement topography map and Dar Zarrouk parameters maps). The maps were interpreted individually by identifying geoelectric parameters favourable to groundwater occurrence. The maps were combined to form a composite entity from which the groundwater potential of the study area was evaluated. The groundwater potential map was used to classify the study area into good, moderate and poor groundwater zones. The hydrogeologic importance of the equivalent current density pseudo-sections, the basement depressions identified on the basement topography map, maps generated from the primary and secondary (Dar Zarrouk) parameters corroborated the deductions from the groundwater map. Zones identified to have moderate and good groundwater potential can be considered for groundwater development at University of Ibadan cooperative housing estate in Alabata, Ibadan. The southern/northern portion was considered as good groundwater prospect area.

Acknowledgements

The authors acknowledged Mr. Isaac O. Babatunde with profound appreciation for the assistance during all the data acquisition. Sincere thanks are given to the anonymous reviewers.

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Professional paper

Received: July 31, 2013
Accepted: November 13, 2013

An attempt to improve geotechnical properties of some highway lateritic soils with lime

Poskus izboljšave geotehničnih lastnosti nekaterih lateritnih tal za ceste z dodajanjem apna

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Abstract

An attempt to stabilize some soils from failed sections of the Sagamu–Papalanto road, southwestern Nigeria with lime was undertaken with a view to improve the geotechnical properties of the soils. The soils were treated with 0 % to 20 % by mass of lime, compacted at the Modified AASHTO level and subjected to consistency limits, unconfined compressive strength (UCS) and California bearing ratio (CBR) tests. Increasing content of lime addition resulted in soils with reducing plasticity with an optimum range of 6 % to 8 % while the UCS and CBR increased. Furthermore addition of between 6 % and 10 % of lime produced soils with desirable strength for use as base course materials. However despite the continuous increase in CBR with increasing lime addition, none of the soils meet the unsoaked CBR requirement for use as base course materials. However the soils qualify for use as subbase materials. Thus, it can be concluded that, the soils responded positively to lime addition; however the degree of response and the eventual effect on its suitability for use varied from soil to soil.

Key words: lime, geotechnical properties of soil, lateritic soils, base course, aggregation

Izvleček

Preizkusili smo možnost izboljšanja geotehničnih lastnosti tal na poškodovanih odsekih ceste Sagamu–Papalanto v jugozahodni Nigeriji z dodajanjem apna. Tlom smo dodajali masni delež apna od 0 % do 20 %, jih stiskali na modificiran AASHTO-nivo in določili njihove konsistenčne meje, nezaprto tlačno trdnost (UCS) in opravili geomehanski CBR-preizkus. Dodajanje apna v razponu od 6 % do 8 % je zmanjšalo plastičnost tal, zvečala sta se UCS in CBR. Z nadaljnjim dodajanjem apna, med 6 % in 10 %, smo dosegli trdnost, ki je potrebna za cestno nosilno plast. Kljub povečanju CBR z dodanim apnom nobena od preiskovanih tal ni zadostila zahtevam neovlaženega CBR. Ugotavljamo, da se dodajanje apna tlom obnese, vendar sta stopnja izboljšanja lastnosti in vpliv na primernost za uporabo odvisna od vrste tal.

Ključne besede: geotehnične lastnosti tal, apno, laterit, nosilna plast tal, struktura tal

Introduction

Frequent failure of structures, particularly roads in Nigeria, leading to loss of lives and properties has necessitated the need to find ways of ensuring the stability of the road pavements. In southwestern Nigeria, lateritic soils which are referred to as tropical red soils are by far the most abundant and most common materials used for road construction works either as subbase or subgrade options. They have found wide application and have been used extensively in construction of dams, embankments as well as buildings. Thus, as opined by Oyediran et al.[1], lateritic soils with appropriate geotechnical properties are indispensable. However, the engineering characteristics of lateritic soils as indicated by Townsend[2], vary considerably, depending on factors such as parent material, climate, topography, drainage, vegetation, age, and they usually form in tropical and other similar hot and humid climatic regions, where heavy rainfall, warm temperatures and good drainage lead to the formation of thick horizons of reddish soil profiles rich in iron and aluminum. CIRIA[3] confirmed that laterite in all its form is a highly weathered natural material formed by the concentration of the hydrated oxides of iron and aluminum. This concentration may be by residual accumulation or by solution, movement and chemical precipitation. Goswami and Mahanta[4] noted that they occur mostly as the capping of hills and therefore provide excellent borrow areas for extensive use in various construction activities. However the relative abundance of lateritic soils notwithstanding, the soils must satisfy requirements for its intended use and when this is not the case the need to seek ways of improving the soil becomes imperative.

Lime stabilization of soils is not new. Several researchers Remus and Davidson[5], Ingles and Metcalf[6], Sherwood[7], Little[8], Bell[9], Rajasekaran and Rao[10], Nalbontoglue and Tuncer[11], Khattab et al.[12], Hebib and Farrell[13], Petry and Glazier[14], Koslanant et al.[15], Khattab et al.[16], James et al.[17], Chen et al.[18] and Harris et al.[19], have worked on lime stabilisation of soils, albeit with temperate soils. As noted by Attoh-Okine[20] however, the geotechnical properties of lateritic soils are quite different from the soils developed under cold or temperate climates. Though, several other authors including Ola[21], Osula[22], Osinubi[23], Galvao et al.[24], Huat et al.[25] and Mohd Yunus et al.[26] have stabilised tropical residual soils with lime, yet these instances with tropical soils, are still very scanty in literature. More work still needs to be done to surmount the barrier of limited information. More so, the results obtained from these studies and those from previously reported work on tropical residual soils show no well defined or uniform trend for the change of some geotechnical properties of these soils upon the addition of lime. These variations and uncertain trends observed may not be unconnected with the individual soil mineralogy and parent material. This investigation is therefore an attempt to determine the effect of addition of varying quantities of lime (0 % to 20 % by weight) on the geotechnical properties of some residual lateritic soils from failed sections of the recently constructed Sagamu-Papalanto road. Furthermore, the optimum content of lime required to produce desired results (improving the soil properties) while achieving better pavement soils will be deduced. Moreover, a significant contribution to the existing literature on lime stabilisation of residual lateritic soils and assessment of its response to varying lime content is expected will be presented.

Study Area

The study area lies within Latitude 3° 12’ to 3° 30’ and longitude 6° 51’ to 6° 54’ and is located in the southwestern part of Nigeria. In terms of Geology the area is underlain by the Sedimentary rocks of southwestern Nigeria (Omatsola and Adegoke[27]) and falls within the Ewekoro formation. The Ewekoro formation which is Paleocene in age is highly fossiliferous and consists of economic deposits of limestone presently quarried by the West African Portland Cement Company in Ewekoro and Sagamu. The general succession of the rock units comprising of the Ewekoro Formation has been described by several authors (Kogbe[28]; Adegoke et al.[29]). Specifically the soils from the sampling points (Figure 1), developed over claystone and shale (Makelu and Ikereku), and shale and limestone (Someke).

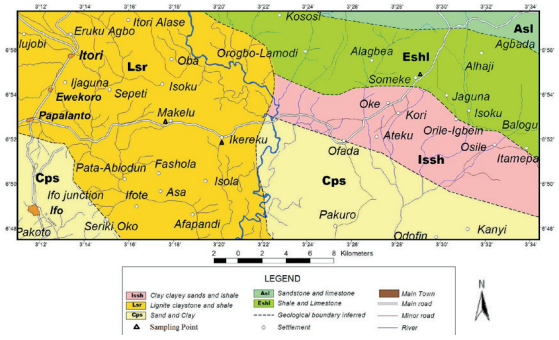

Figure 1: *Geological map of the study area showing sampling points.*

Materials and Methods

Three bulk residual lateritic soil samples were obtained at depths between 0.5 m and 1.0 m from borrow pits at failed sections along the recently constructed Sagamu–Papalanto road for this work. The choice of the sampling points was guided by proximity to failed portions on the road. The lateritic soils were air dried for two weeks prior to laboratory analyses and later subjected to geotechnical tests for the determination of grain size distribution, consistency limits, California bearing ratio (CBR) and unconfined compressive strength (UCS). The geotechnical tests were done in accordance with Bs1377[30] test procedures with some slight modification in some cases to accommodate the lateritic nature of the soils. For example, wet sieving was used for the determination of particle size distribution to ensure effective detachment of the fine grained particles from the coarse grained particles. About 500 g of air-dried soil mixed with distilled water and calgon (deflocculating agent) was stirred for about 20 min for effective dispersal of the soil grains. The wet suspension was then passed through a 63 μm sieve to separate the coarse fraction from the fines fraction. The coarse fraction retained on the sieve was then oven dried, allowed to cool and sieved through a set of sieves. The fraction retained on each sieve was eventually weighed. The fines fraction which passed through the 63 μm sieve was separated into silt and clay size fractions using sedimentation analysis based on Stokes law. CBR tests were performed on compacted samples in both unsoaked and soaked conditions Soaking of the samples in water was done for 24 h in accordance with the Nigerian general specification (FMWH, 1997[31]) before the determination of the soaked CBR to simulate natural conditions and assess the extent to which the ingress of water would expand and weaken the soils. For completeness of investigation effective segregation of soil grains was achieved through constant agitation. Varying quantities of (2, 4, 6, 8, 10, and 20) % of lime (mass fractions) was added to soil samples and mixed thoroughly to allow for intimate mixing. The mixed soil samples were left for 48 h to cure and mellow and subsequently remixed to achieve a homogenous mix prior to compaction at Modified AASHTO level of compaction. The Modified AASHTO compaction was desirable because it is usually achievable with conventional field equipment. In the determination of the UCS all the samples were cured for 7 d and were compacted at OMC to simulate field moisture compaction conditions. The bulk chemical composition of the soils was determined with use of atomic absorption spectrophotometer (AAS) technique which involved the use of air-dried ground soil weighed and placed in an Erlenmeyer flask with the addition of 0.05N HCl + 0.025N H_2SO_4 as the extracting solution. The samples were subsequently placed in a mechanical shaker and then filtered. The extract was then analyzed directly for the determination of concentration of elements using atomic absorption. Furthermore the mineralogical composition of the soils was determined using x-ray diffraction (XRD). Powdered samples of the soil were pelletized and sieved to 0.074 mm. These were later mixed with acetone to produce a thin slurry and each sample mixture was applied to a glass was scanned through the Siemens D500 Diffractometer (using MDI Data Scan and JADE 8 softwares) for the determination of XRD.

Results and Discussion

Soil Properties

The particle size distribution of the studied soils is summarized in Table 1 and the grading curves displayed on Figure 2. The summary shows that Makelu soil contained the highest amount of clay size fraction (12.0 %), Someke soils contained the highest amount of silt size

(57.0 %) and amounts of fines (65.0 %) fraction while the Ikereku soils possessed the highest amount of gravel size (14.0 %) and sand size (43.0 %) fractions. The grading curves shows all the soils are well graded and hence will be expected to compact to a lower porosity and permeability than uniformly graded soils (Oyediran and Adeyemi[32]). However as indicated by Oyediran and Williams[33], soils with amounts of fines less than 50 % are expected to possess better engineering properties while those with amounts of fines greater than 50 % are expected to pose field compaction problems when used either as base course or subbase materials. Hence on the basis of amounts of fines Someke and Makelu soils are not suitable for use as subbase or base course materials in the construction of roads as they will pose problems.

Furthermore none of the soils satisfy the requirements of the Nigerian Federal Ministry of Works and Housing (FMWH[31]) specification (amounts of fines 5–15 % for base-course materials) for highway construction. Thus, the materials do not qualify for use as base-course materials. The AASHTO classification of the soils also shows that they fall in the A-6 and A-7-6 subgroup, which indicates that the materials are fair to poor subgrade soils. These characteristics displayed by the soils, may in part be responsible for the failures noticed on the road sections.

In terms of consistency, Casagrande chart (Figure 3) classification shows the soils are all inorganic soils of medium plasticity and hence compressibility. All the soils fall above the A-Line possibly indicating close or similar clay mineralogy. The soils are expected to undergo

Table 1: *Index, chemical and mineralogical properties of studied soils*

Parameter (%)	Sample	Someke	Makelu	Ikereku
Particle Size Distribution	Gravel	8.0	8.0	14.0
	Sand	27.0	35.0	43.0
	Clay	8.0	12.0	7.0
	Silt	57.0	45.0	36.0
	Fines	65.0	57.0	43.0
Consistency Limits	Liquid Limit	33.0	44.0	32.0
	Plastic Limit	14.0	18.0	20.0
	Plasticity Index	19.0	26.0	12.0
Classification	Casagrande Classification	Medium Plasticity and Compressibility		
	AASHTO Classification	A-6	A-7-6	A-6
Bulk Chemical Composition	Al_2O_3	1.71	21.86	15.96
	CaO	0.57	0.10	0.32
	Fe_2O_3	5.73	10.01	6.05
	K_2O	1.87	0.19	2.19
	MgO	0.42	0.18	0.57
	Na_2O	0.24	0.04	0.35
	P_2O_5	0.02	0.08	0.04
	SiO_2	88.21	66.11	73.18
	TiO_2	1.23	1.43	1.34
	Silica sesqui-oxide ratio	27.95	3.98	6.28
Mineralogical Composition	Quartz	51.18	61.73	61.38
	Kaolinite	27.97	18.75	31.53
	Labradorite	20.85	19.52	-
	Haematite	-	-	7.09

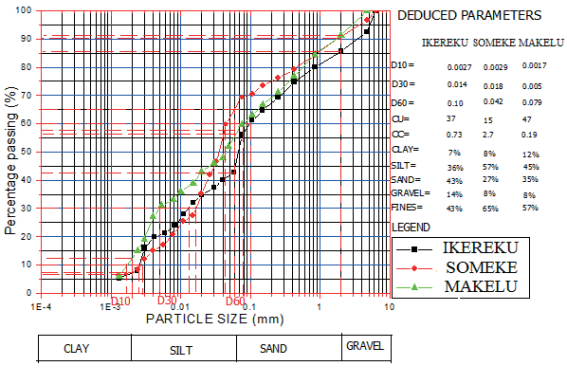

Figure 2: *Grading curves of studied soils.*

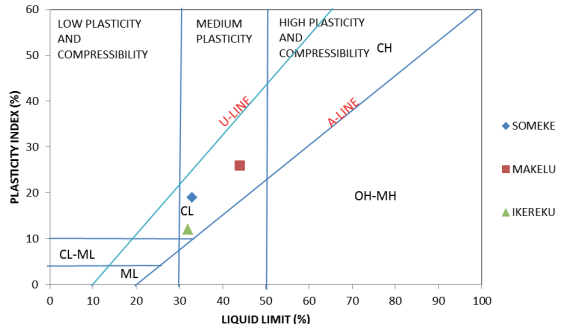

Figure 3: *Casagrande chart classification of studied soils.*

moderate swelling and shrinkage when loaded as a result of their medium plasticity. According to FMWH[31], soils suitable for use as base course materials must possess liquid limit and plasticity index values < 30 % and < 13 % respectively. All the soils have liquid limits greater than 30 % and all except the Ikereku soil (12 %) possess plasticity index greater than 13 % and hence will not perform creditably well as base course materials. However for use as subbase materials, suitable soils must display liquid limit and plasticity index < 35 % and < 16 % respectively. It can be concluded that only the Ikereku soil meets both conditions of this requirement.

The chemical composition of the soils indicate that all the soils contain high amounts of SiO_2 with the Someke soils having the highest (88.21 %) and the Makelu soil possessing the lowest (66.11 %). The Someke soil also possesses the highest silica sesquioxide ratio (27.95 %) and Makelu soil the lowest (3.92 %). However a reverse trend was noticed with the Makelu soil having the highest amounts of Al_2O_3 (21.86 %) and Fe_2O_3 (10.01 %) while Someke soil has the lowest Al_2O_3 (1.71 %) and Fe_2O_3 (5.73 %). Moh[34], Ola[21, 35] and Anifowose[36] have shown that soil type and its composition influence the results of stabilisation. It should be noted that Makelu soil has the highest clay content, plasticity values, Fe_2O_3 and Al_2O_3 content. The x-ray diffraction analysis further revealed that quartz is the most abundant mineral in all the soils. All the soils contain kaolinite as the clay mineral with the Makelu soil having the lowest amount (18.75 %). Minor amounts of Hematite (7.09 %) were observed in the Ikereku soil while the Someke and Makelu soils possess 20.85 % and 19.52 % of labradorite respectively.

Effect of Lime on Consistency limits

The consistency limits of the soils in response to lime stabilisation are presented in Table 2, while the pictorial variations are displayed on Figures 4, 5 and 6. There was a reduction in liquid limit (LL) of all the soils as lime content increased from 0 to 20 %. The same trend was observed for the plastic limit (PL) of all the soils. The highest reduction in LL and PL was observed at the addition of 20 % by weight of lime. Makelu and Ikereku soils showed a uniform trend of continuous reduction in plasticity with increase in lime content. The PI reduced steadily with the highest change of 12 % and 14 % respectively for Makelu and Ikereku soils with the addition of 20 % lime. The immediate impact of lime on the soils leading to reduction in plasticity with lime addition is attributed to cation exchange and aggregation.

The reactions take place rapidly and produce immediate improvements in soil plasticity (reduced plasticity and shrink/swelling potential) and workability. Initially the water content is reduced followed by flocculation and agglomeration of clay particles which brings about textural change which lead to eventual decrease in PI and increase in workability (Terrel et al.[37]). The flocculation and agglomeration are caused by the increased electrolyte content of the pore water and as a result of ion exchange by the clay to the calcium form. The net result of cation exchange and flocculation–agglomeration is soil modification (Little[38]). This brings about substantial reduction and stabilization of the adsorbed water layer, increased internal friction among the agglomerates and greater aggregate shear strength and finally much greater work-

Table 2: *Consistency limits of soil-lime mix*

Sample	Someke				Makelu				Ikereku			
Lime (%)	LL (%)	PL (%)	PI (%)	Change in PI (%)	LL (%)	PL (%)	PI (%)	Change in PI (%)	LL (%)	PL (%)	PI (%)	Change in PI (%)
0	33.0	14.0	19.0	-	44.0	18.0	26.0	-	32.0	20.0	12.0	-
2	30.0	13.8	17.0	–11	42.9	17.5	25.4	–2	30.8	19.4	11.4	–5
4	29.3	13.3	16.0	–13	41.5	16.5	25.0	–4	29.7	18.7	11.0	–8
6	28.2	12.9	15.3	–19	40.6	15.9	24.7	–5	29.5	18.6	10.9	–9
8	27.8	12.8	15.0	–21	39.3	14.9	24.4	–6	27.9	17.2	10.7	–11
10	27.2	11.0	16.2	–15	35.2	12.0	23.2	–11	27.6	17.1	10.5	–13
20	26.0	10.1	15.9	–16	34.9	11.9	23.0	–12	26.3	16.0	10.3	–14

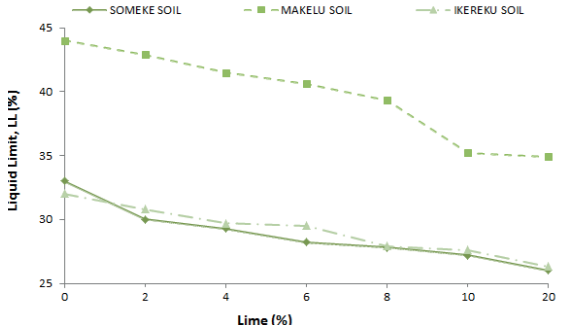

Figure 4: *Variation in liquid limit of studied soils.*

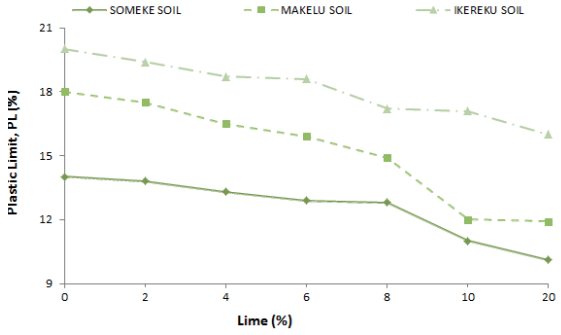

Figure 5: *Variation in plastic limit of studied soils.*

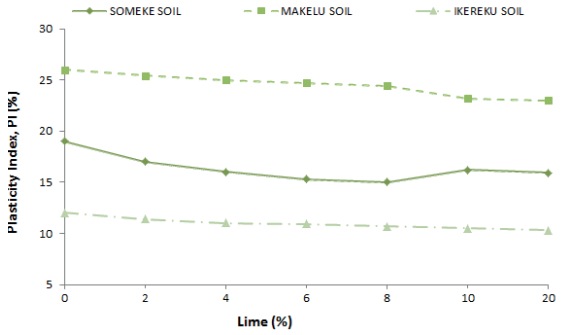

Figure 6: *Variation in plasticity index of studied soils.*

ability due to the textural change from plastic clay to a friable, sand-like material.

According to Townsend et al.[39], the principal components of tropical lateritic soils that are responsible for pozzolanic reactions are amorphous silica and alumina. It is believed that clay minerals that are usually found in tropical residual soils such as kaolinite, halloysite, and crystallized aluminum hydroxides also contribute to the pozzolanic reactions, while iron compounds are considered harmful or neutral. Little and Shafee Yusuf[40], did indicate that the reactivity of lime with soil is predicated on the type and the amount of clay minerals present in the soil. However in terms of plasticity index (PI), Someke soil responded to lime addition with an initial reduction up to 8 % lime addition. Upon addition of 10 % by weight of lime the PI increased but later reduced when the lime content was increased to 20 %. This reaction may not be unconnected with the high sesqui-oxide ratio of the Someke soil due to the fact that increase in sesqui-oxides results in decrease in cation exchange capacity and moisture retentivity of soil.

The Ikereku and Someke soils changed from medium plasticity soils to low plasticity soils while the soil from Makelu also recorded 12 % reduction in plasticity with 20 % lime addition. The reduction in plasticity (from medium to low) results in soils with low swelling and shrinkage potential. As indicated by Little[8], soil swell potential and swelling pressure are normally significantly reduced by lime treatment. Furthermore, the reduction in PI associated

with virtually all fine-grained soils upon the addition of lime is a significant indication of the reduction of swell potential due to lime stabilization. The reduction in PI of these soils compares well with findings of Ola[35], Anifowose[36], Osula[22], and Osinubi[23] who worked on lime-soil mixtures. It must however be noted that despite the reduction in plasticity occasioned by increasing lime content, Makelu soil still did not meet the requirement for use as either base course or subbase course material. However Someke and Ikereku soils can be said to have met the requirements for use as subbase course materials.

Effect of Lime on Strength parameters

The Unconfined Compressive Strength (UCS) of the studied soils in response to lime addition is displayed on Table 3 and shown for clarity with Figure 7. The UCS of the soils increased with the addition of lime from 0 % to 20 %. It was observed that on initial addition of lime up to 6 % there was a change of about 100 % in UCS for all the soils. Further increase in lime addition brought an exponential increase in UCS between 274 % and 503 %. 20 % addition of lime resulted in maximum increase in UCS with Someke soil showing the greatest response with a 503 % increase in UCS. The addition of between 6 % and 10 % by mass of lime produced soils (Makelu and Ikereku) with desirable strength which meet the requirements (FMWH[31]) of > 103 kN/m^2 for use as subgrade materials. Results obtained from this study are similar to that which occurs in soil-lime mixtures with an immediate cation exchange reaction, followed by a time-dependent pozzolanic reaction, during which strength is developed. The UCS gain of the lime-treated soil may not be unconnected to the formation of calcium aluminate hydrate as a result of the pozzolanic reaction between lime and kaolinite (Osinubi[41]). This assertion is further supported by Little[42] who showed on the basis of the Energy Dispersive X-Ray (EDX) test analysis that the pozzolanic reaction had already converted some of the clay minerals to (calcium silicate hydrate) CSH after a cure period of 7 d.

These improvements are largely the result of the flocculated particle structure and cementation process that must have taken place. The addition of lime increases the soil strength thereby increasing the mobility of wheeled vehicles involved in construction operations and they help provide a stable working platform for all construction equipment. An increase in load bearing capabilities of the subgrade and continuous increase results in increased longevity of roadway by continued strength gains of the treated soil, creating a permanent pavement foundation.

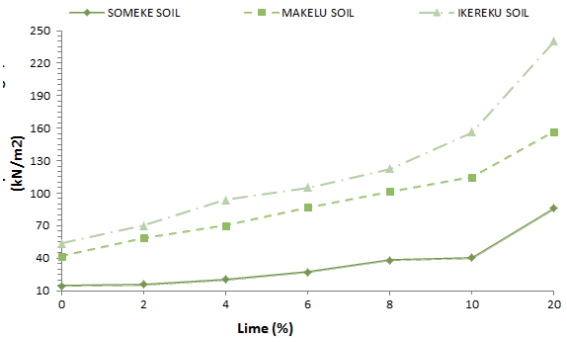

Figure 7: *Variation in Unconfined Compressive Strength (UCS) of studied soils.*

Table 3: *Unconfined Compressive Strength (UCS) of soil-lime mix*

Sample	Someke		Makelu		Ikereku	
Lime (%)	UCS (kN/m^2)	Change (%)	UCS (kN/m^2)	Change (%)	UCS (kN/m^2)	Change (%)
0	14.3	-	41.9	-	53.7	-
2	15.8	10	58.6	40	70.0	30
4	20.2	41	70.0	67	93.6	74
6	27.1	90	86.7	107	105.4	96
8	37.9	165	101.5	142	122.7	128
10	40.4	183	114.8	174	156.2	191
20	86.2	503	156.7	274	240.4	348

The effect of lime addition on the CBR (Table 4 and Figures 8 and 9) shows an increase in CBR for all the soils at both soaked and unsoaked conditions. Maximum increase of up to 33 % and 40 % were achieved for the soils respectively under unsoaked and soaked conditions. The increase in CBR is thought to be due to the formation of various cementing agents due to pozzolanic reaction between silica present in the soil and lime. The effect of soaking, though very marginal was observed particularly for the Someke soil. The difference in CBR between soaked and unsoaked soils is as a result of water absorption which further weakened the soil. Despite the continuous increase in CBR with increasing lime addition, none of the soils meet the ≥ 80 % unsoaked CBR (FMWH[31]) for soils that can be used as base course materials. However the soils qualify for use as sub base materials. Thus from the results, the strength characteristics were improved with increasing lime content.

Conclusions

An attempt to improve some highway lateritic soils from failed sections of the Sagamu -Papalanto road has led to the following conclusions;

— All the soils studied responded positively to lime addition; however the degree of response and the eventual effect on its suitability for use varied from soil to soil.

— There was a reduction in liquid limit (LL) and plastic limit (PL) of all the soils as lime content increased from 0 % to 20 %, with the highest reduction observed on addition of 20 % lime. Makelu and Ikereku soils showed a uniform trend of continuous reduction in plasticity with increase in lime content as the PI reduced steadily with the highest change of 12 % and 14 % respectively for the soils with the addition of 20 % lime. Reduction in PI is expected to result in increased workability of the soils.

Table 4: *California Bearing Ratio (CBR) of soil-lime mix*

Sample	Someke				Makelu				Ikereku			
	Soaked		Unsoaked		Soaked		Unsoaked		Soaked		Unsoaked	
Lime (%)	CBR (%)	Change (%)	CBR (%)	Change (%)	CBR (%)	Change (%)	CBR (%)	Change (%)	CBR (%)	Change (%)	CBR (%)	Change (%)
0	22.6	-	22.9	-	23.7	-	32.1	-	17.4	-	27.9	-
2	23.9	7	24.4	7	24.4	3	33.1	3	18.1	4	28.4	2
4	24.7	8	24.8	8	25.7	8	36.5	14	19.7	13	29.2	5
6	26.0	16	26.5	16	27.0	14	36.7	14	20.5	18	29.9	7
8	27.0	23	28.1	23	28.6	21	37.9	18	22.1	27	31.0	11
10	28.4	27	29.1	27	29.9	26	38.1	19	23.1	33	32.0	15
20	29.1	33	30.5	33	31.5	33	38.9	21	24.4	40	33.1	19

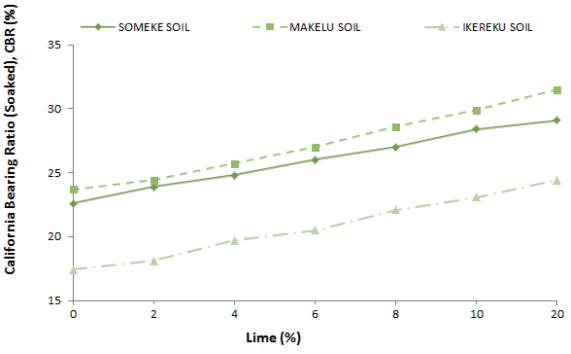

Figure 8: *Variation in soaked California Bearing Ratio (CBR) of studied soils.*

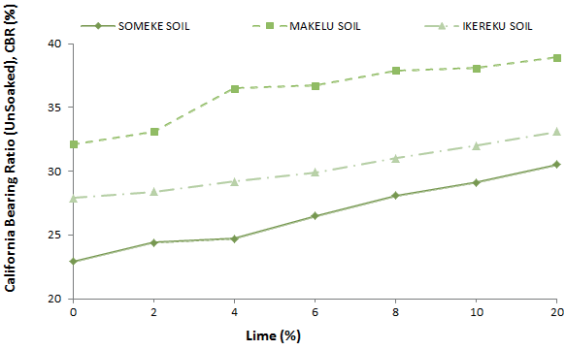

Figure 9: *Variation in unsoaked California Bearing Ratio (CBR) of studied soils.*

— The plasticity index of the Someke soil increased on addition of 10 % by weight of lime after initial steady and continuous decrease between 2 % and 8 % lime addition. It is thus safely assumed that for all the soils, 6 % to 8 % lime content which produced a decrease of 21 %, 6 % and 11 % in PI respectively for Someke, Makelu and Ikereku soils is the optimum range of lime addition. Addition of lime reduced the plasticity of the soils from medium to low as observed in the Casagrande chart classification hence producing soils with low swelling and shrinkage potential.
— The UCS of all the soils increased with increasing lime content with a maximum increase of 503 % (Someke soil) on addition of 20 % lime. It was observed that on initial addition of lime up to 6 % there was a change of about 100 % in UCS for all the soils. The addition of between 6 % and 10 % by weight of lime produced soils (MAKELU and IKEREKU) with desirable strength for use as base course materials.
— The unsoaked and soaked CBR of all the soils increased with increasing lime content respectively up to 33 % and 40 %. Despite the continuous increase in CBR with increasing lime addition, none of the soils meet the unsoaked CBR requirement for use as base course materials. At best, however the soils qualify for use as sub base materials.

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— Poljudni članki, ki povzemajo znanstvene in strokovne dogodke zavzemajo do dve (2) strani.

Recenzentski postopek

Vsi prispevki bodo predloženi v recenzijo. Recenzent oceni primernost prispevka za objavo in lahko predlaga kot pogoj za objavo dopolnilo k prispevku. Recenzenta izbere uredništvo med strokovnjaki, ki so dejavni na sorodnih področjih, kot jih obravnava prispevek. Avtorji morajo predlagati tri recenzente. Pravico imajo predlagati ime recenzenta, za katerega ne želijo, da bi recenziral njihov prispevek. Uredništvo si pridržuje pravico, da izbere druge recenzente. Recenzent ostane anonimen. Prispevki bodo tudi tehnično ocenjeni in avtorji so dolžni popraviti pomanjkljivosti. Končno odločitev za objavo da glavni urednik.

Oblika prispevka

Prispevek lahko posredujte preko e-pošte ter na USB mediju ali CD-ju.
Predloga za pisanje članka se nahaja na spletni strani: **www.rmz-mg.com**.
Besedilo naj bo podano v urejevalniku besedil Word. Digitalni zapis naj bo povsem enostaven, brez zapletenega oblikovanja, deljenja besed, podčrtavanja. Avtor naj označi le krepko in kurzivno poudarjanje.

Zgradba prispevka

Naslov

Naslov članka naj bo natančen in informativen in naj ne presega 100 znakov. Avtor naj navede tudi skrajšan naslov članka. Naslov članka je podan v angleškem in slovenskem jeziku.

Information on the Authors
Information on the authors should include the first and last name of the authors, the address of the institution and the e-mail address of the leading author.

Abstract
The abstract presenting the purpose of the article and the main results and conclusions should contain no more than 180 words. It should be written in Slovene and English.

Key words
A list of up to 5 key words (3 to 5) that will be useful for indexing or searching. They should be written in Slovene and English.

Introduction

Materials and methods

Results and discussion

Conclusions

Acknowledgements

References
The sources should be cited in the same order as they appear in the article. They should be numbered with numbers in square brackets. Sources should be cited according to the SIST ISO 690:1996 standards.

Monograph:
[1] Trček, B. (2001): *Solute transport monitoring in the unsaturated zone of the karst aquifer by natural tracers*. Ph. D. Thesis. Ljubljana: University of Ljubljana 2001; 125 p.

Journal article:
[2] Higashitani, K., Iseri, H., Okuhara, K., Hatade, S. (1995): Magnetic Effects on Zeta Potential and Diffusivity of Nonmagnetic Particles. *Journal of Colloid and Interface Science*, 172, pp. 383–388.

Electronic source:
CASREACT – Chemical reactions database [online]. Chemical Abstracts Service, 2000, renewed 2/15/2000 [cited 2/25/2000]. Available on: <http://www.cas.org/casreact.html>.

Podatki o avtorjih
Podatki o avtorjih naj vsebujejo imena in priimke avtorjev, naslov pripadajoče inštitucije ter elektronski naslov vodilnega avtorja.

Izvleček
Izvleček namena članka ter ključnih rezultatov z ugotovitvami naj obsega največ 180 besed. Izvleček je podan v angleškem in slovenskem jeziku.

Ključne besede
Seznam največ 5 ključnih besed (3–5) za pomoč pri indeksiranju ali iskanju. Ključne besede so podane v angleškem in slovenskem jeziku.

Uvod

Materiali in metode

Rezultati in razprava

Sklepi

Zahvala

Viri
Uporabljane literaturne vire navajajte po vrstnem redu, kot se pojavljajo v prispevku. Označite jih s številkami v oglatem oklepaju. Literatura naj se navaja v skladu s standardom SIST ISO 690:1996.

Monografija:
[1] Trček, B. (2001): *Solute transport monitoring in the unsaturated zone of the karst aquifer by natural tracers*. doktorska disertacija. Ljubljana: Univerza v Ljubljani 2001; 125 str.

Članek v reviji:
[2] Higashitani, K., Iseri, H., Okuhara, K., Hatade, S. (1995): Magnetic Effects on Zeta Potential and Diffusivity of Nonmagnetic Particles. *Journal of Colloid and Interface Science*, 172, str. 383–388.

Spletna stran:
CASREACT – Chemical reactions database [online]. Chemical Abstracts Service, 2000, obnovljeno 15. 2. 2000 [citirano 25. 2. 2000]. Dostopno na svetovnem spletu: <http://www.cas.org/casreact.html>.

Scientific articles, review papers, preliminary notes and professional papers are published in English. Only professional papers will exceptionally be published in Slovene.

Annexes

Annexes are images, spreadsheets, tables, and mathematical and chemical formulas.
Annexes should be included in the text at the appropriate place, and they should also be submitted as a separate document, i.e. separated from the text in the article.
Annexes should be originals, made in an electronic form (Microsoft Excel, Adobe Illustrator, Inkscape, AutoCad, etc.) and in .eps, .tif or .jpg format with a resolution of at least 300 dpi.
The width of the annex should be at least 152 mm. They should be named the same as in the article (Figure 1, Table 1).
The text in the annexes should be written in typeface Arial Regular (6 pt).
The title of the image (also schemes, charts and graphs) should be indicated in the description of the image.
When formatting spreadsheets and tables in text editors, tabs, and not spaces, should be used to separate columns. Each formula should have its number written in round brackets on its right side.
References of the annexes in the text should be as follows: "Figure 1…" and not "as shown below:". This is due to the fact that for technical reasons the annex cannot always be placed at the exact specific place in the article.

Manuscript Submission

Contributions should be sent to the following e-mail address: rmz-mg@ntf.uni-lj.si.

In case of submission on CD or USB flash drive, contributions can be sent by registered mail to the address of the editorial board:
RMZ – Materials and Geoenvironment, Aškerčeva 12, 1000 Ljubljana, Slovenia.

The contributions can also be handed in at the reception of the Faculty of Natural Sciences and Engineering (ground floor), Aškerčeva 12, 1000 Ljubljana, Slovenia with the heading "for RMZ – M&G".

Znanstveni, pregledni in strokovni članki ter predhodne objave se objavijo v angleškem jeziku. Izjemoma se strokovni članek objavi v slovenskem jeziku.

Priloge

K prilogam prištevamo slikovno gradivo, preglednice in tabele ter matematične in kemijske formule.
Priloge naj bodo vključene v besedilu, kjer se jim odredi okvirno mesto. Hkrati jih je potrebno priložiti tudi kot samostojno datoteko, ločeno od besedila v članku.
Priloge morajo biti izvirne, narejene v računalniški obliki (Microsoft Excel, Adobe Illustrator, Inkscape, AutoCad ...) in shranjene kot .eps, .tif ali .jpg v ločljivosti vsaj 300 dpi. Širina priloge naj bo najmanj 152 mm. Datoteke je potrebno poimenovati, tako kot so poimenovane v besedilu (Slika 1, Preglednica 1).
Za besedilo v prilogi naj bo uporabljena pisava Arial navadna različica (6 pt).
Naslov slikovnega gradiva, sem prištevamo tudi sheme, grafikone in diagrame, naj bo podan v opisu slike.
Pri urejevanju preglednic/tabel, v urejevalniku besedila, se za ločevanje stolpcev uporabijo tabulatorji in ne presledki.
Vsaka formula naj ima zaporedno številko zapisano v okroglem oklepaju na desni strani.
V besedilu se je potrebno sklicevati na prilogo na način: „Slika 1 ...", in ne „... kot je spodaj prikazano:" saj zaradi tehničnih razlogov priloge ni vedno mogoče postaviti na točno določeno mesto v članku.

Oddaja članka

Prispevke lahko pošljete po elektronski pošti na naslov rmz-mg@ntf.uni-lj.si.

V primeru oddaje prispevka na CD- ali USB-mediju le-te pošljite priporočeno na naslov uredništva:
RMZ – Materials and Geoenvironment, Aškerčeva 12, 1000 Ljubljana, Slovenija

ali jih oddajte na:
recepciji Naravoslovnotehniške fakultete (pritličje), Aškerčeva 12, 1000 Ljubljana, Slovenija s pripisom „za RMZ – M&G".

The electronic medium should clearly be marked with the name of the leading author, the beginning of the title and the date of the submission to the Editorial Office of RMZ – M&G.

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These instructions are valid from July 2013.

Elektronski mediji morajo biti jasno označeni z imenom vsaj prvega avtorja, začetkom naslova in datumom izročitve uredništvu RMZ – M&G.

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Navodila veljajo od julija 2013.

ISSN 1408-7073