Supplementary online material

Comparison of the NMR and the Acid Value Determination Methods for Quality Control of Input Polysorbates

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^{1}H NMR

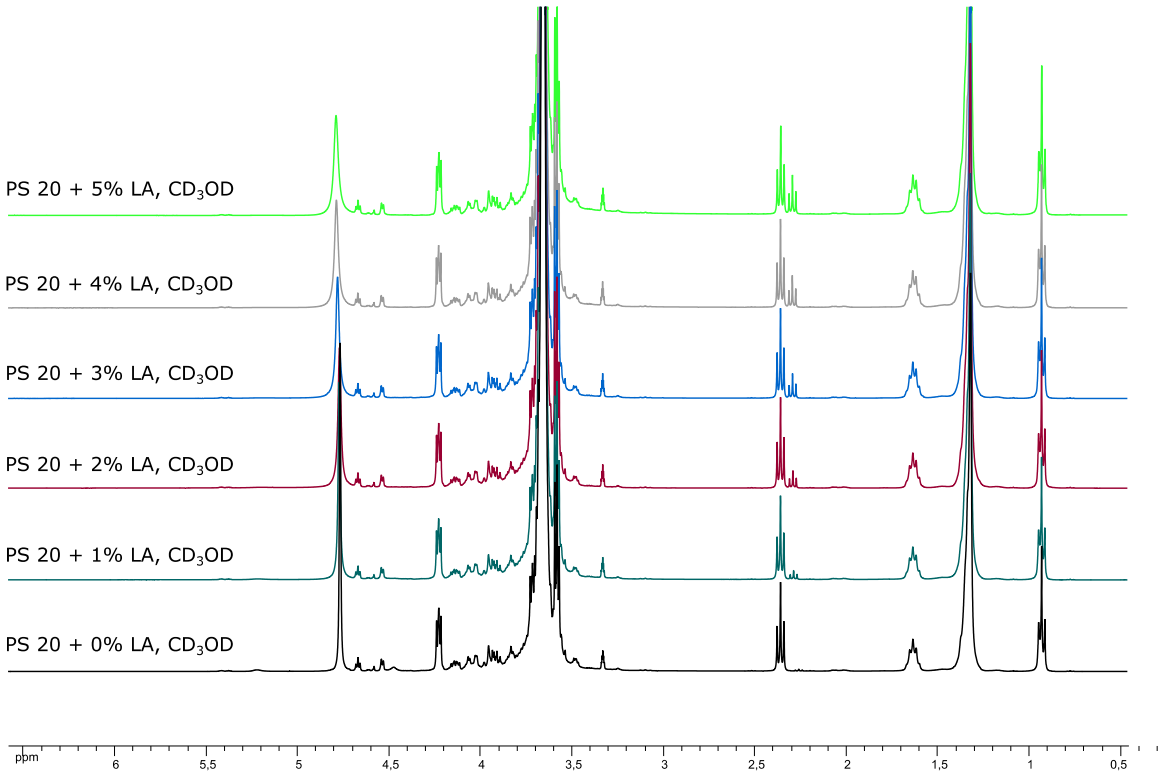

Fig S1. ^{1}H NMR spectra of PS 20 samples with LA spiking, recorded in CD_3OD.

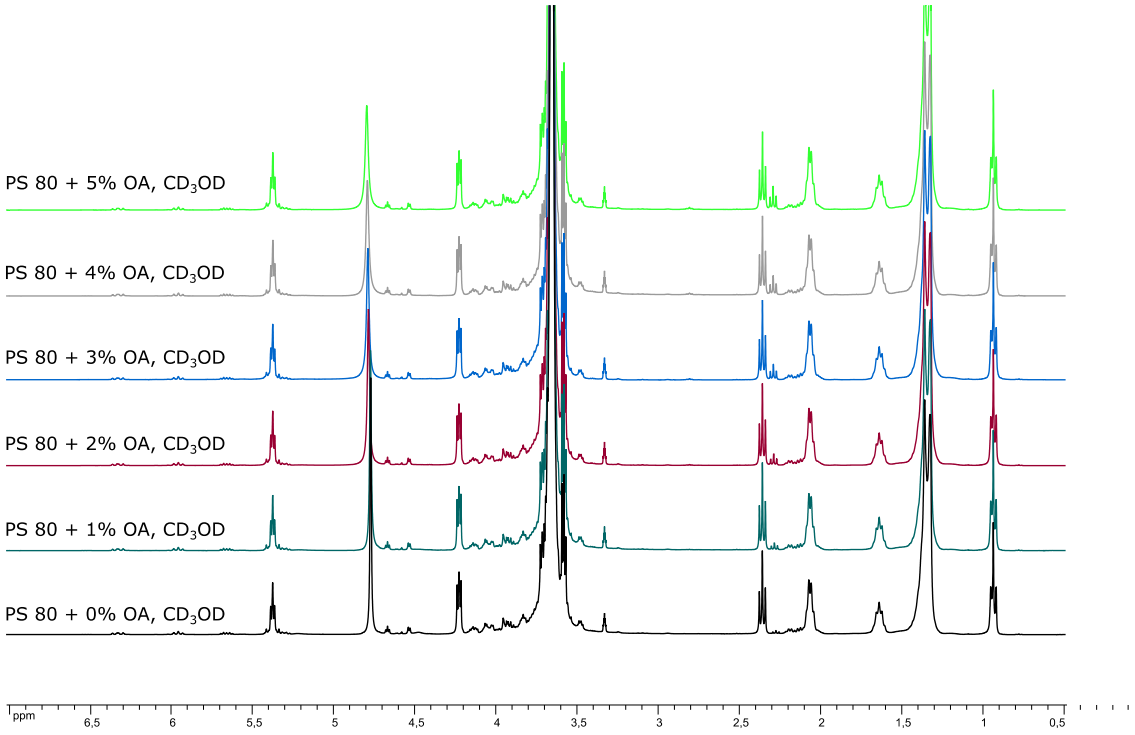

Fig S2. 1H NMR spectra of PS 80 samples with OA spiking, recorded in CD_3OD.

Solvent comparison

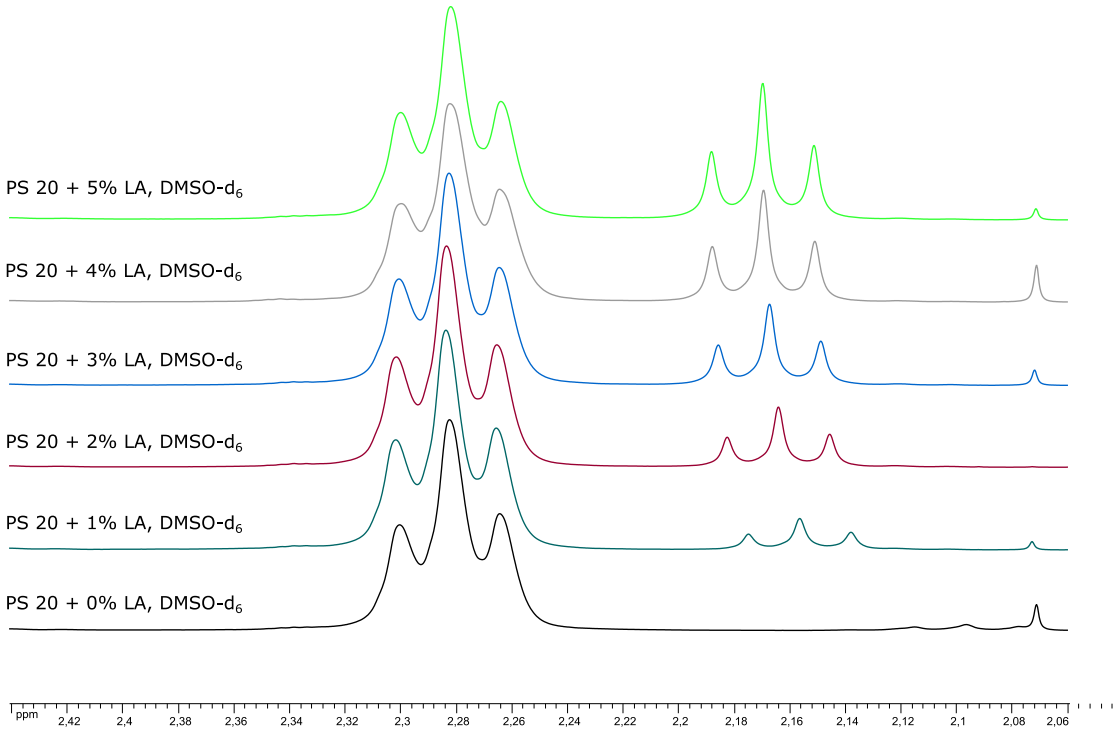

Fig S3. ^{1}H NMR spectra of PS 20 samples, recorded in DMSO-d_6.

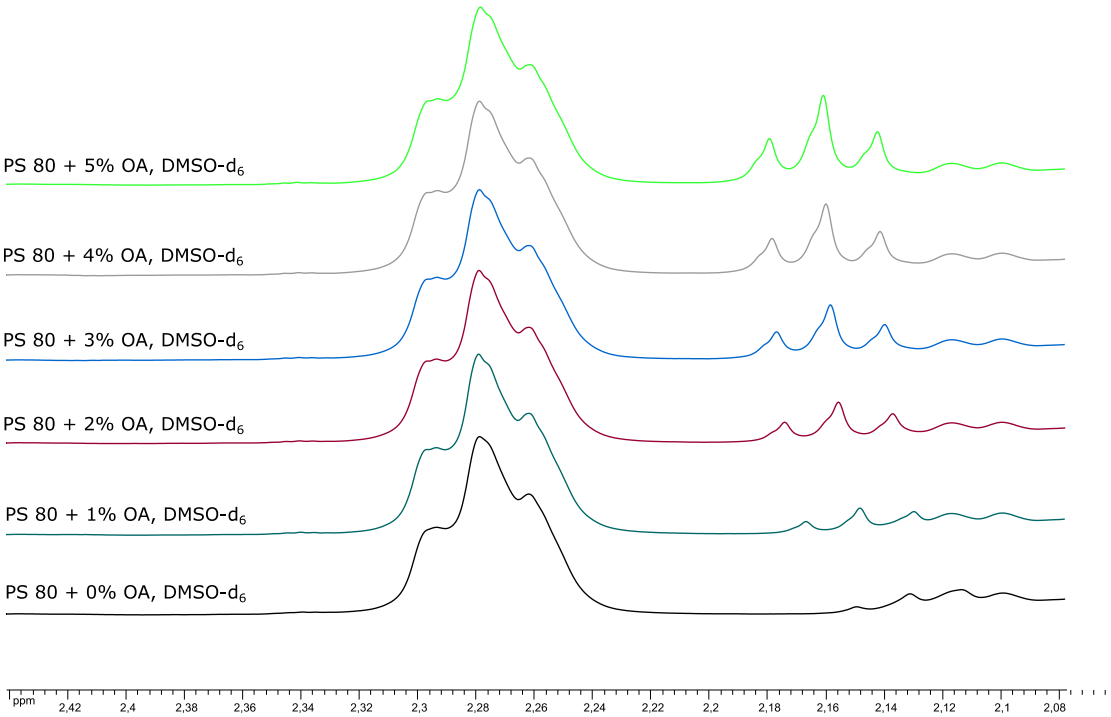

Fig S4. ^{1}H NMR spectra of PS 80 samples, recorded in DMSO-d_6.

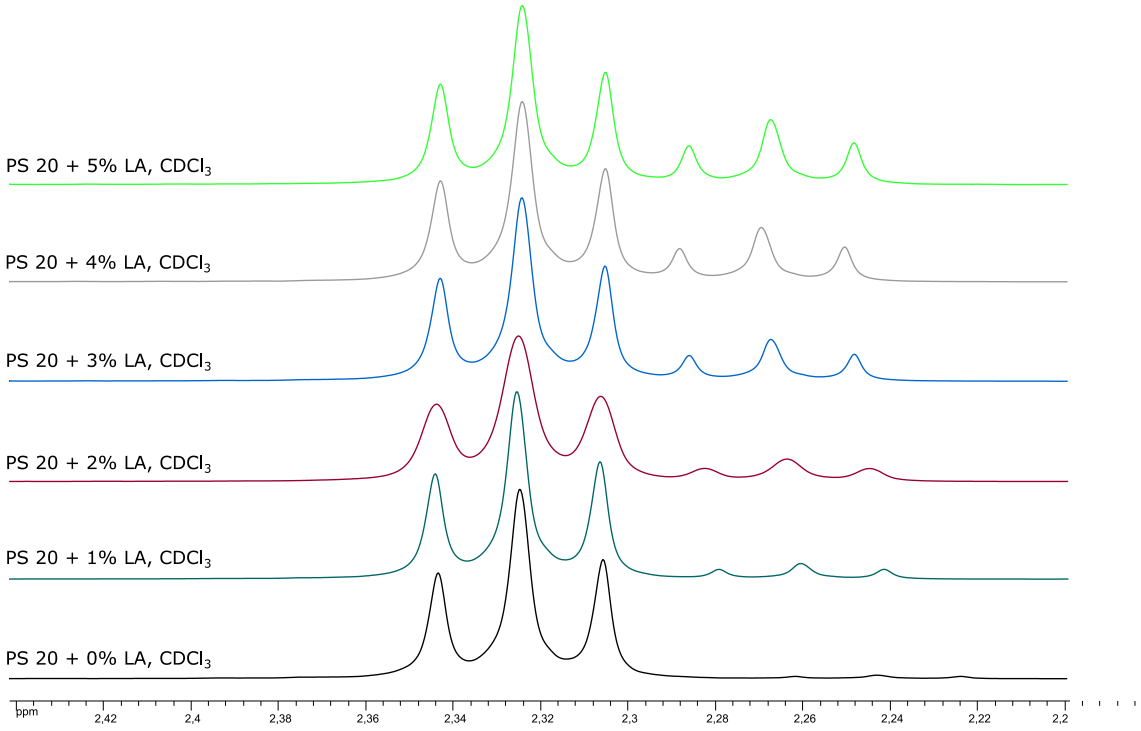

Fig S5. ^{1}H NMR spectra of PS 20 samples, recorded in $CDCl_3$.

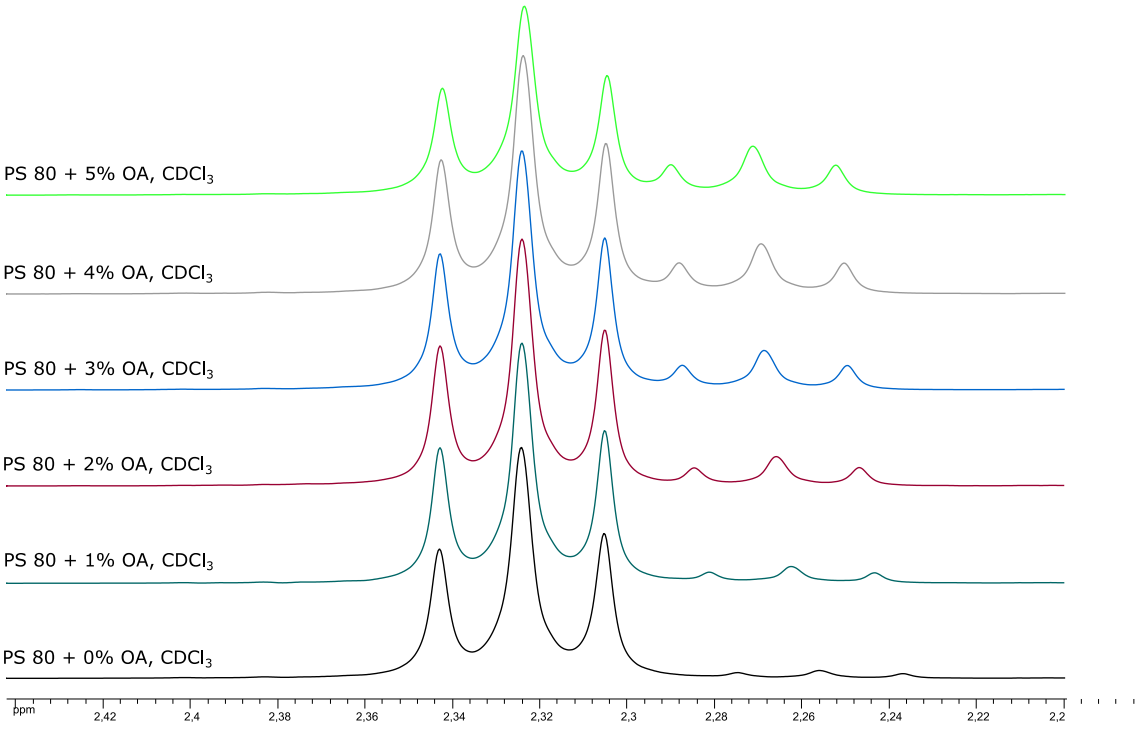

Fig S6. ^{1}H NMR spectra of PS 80 samples, recorded in $CDCl_3$.

Free fatty acid determination

Equation S1. Calculation of the proportion of FFA molecules relative to the total content of FA molecules [%].

$$\% \, \boldsymbol{FA} = \left(\frac{\int_{\boldsymbol{FFA}}}{\int_{\boldsymbol{FFA}} + \int_{\boldsymbol{estrified\ FA}}} \right) \cdot \mathbf{100}$$

∫ FFA = integral value for free fatty acids [/]
∫ esterified FA = integral value for esterified fatty acids = 1 [/]

Table S1. Values of FFAs ∫ and of the proportions of FFA in samples of PS 20 with different levels of LA spiking.

% spiked FA in PS 20	∫ for FFAs	% FFAs
0.00%	0.052	4.94
1.02%	0.113	10.15
2.03%	0.170	14.53
3.01%	0.239	19.29
4.02%	0.313	23.84
4.99%	0.393	28.21

Table S2. Values of FFAs ∫ and of the proportions of FFA in samples of PS 80 with different levels of OA spiking.

% spiked FA PS 80	∫ for FFAs	% FFAs
0.00%	0.097	8.84
0.81%	0.131	11.58
1.98%	0.161	13.87
3.01%	0.193	16.18
3.98%	0.257	20.45
5.02%	0.294	22.72

Acid value determination

Equation S2. Values of I_A were calculated using the following equation, considering the mass of weighed samples and the consumption of 0.1 M NaOH solution.

$$\boldsymbol{I_A} = \frac{\mathbf{5.610} \cdot \boldsymbol{V_{NaOH}} \cdot \boldsymbol{f}}{\boldsymbol{m}}$$

V_{NaOH} = volume of 0.1 M NaOH solution [mL]
f = factor of the volumetric NaOH solution [/]
m = weight of the individual sample [g]

When calculating values of I_A, the factor of the volumetric NaOH solution that represents the ratio between the prepared and the required concentration of the NaOH solution was taken into account. Three repetitions of the determination of I_A for each sample were carried out and the average of three measurements taken.

Table S3. I_A values of the PS 20 samples with LA spiking.

% spiked LA in PS 20	**I_A**
0.00%	0.80
1.02%	3.6
2.03%	6.5
3.01%	9.2
4.02%	12.0
4.99%	14.7

Table S4. I_A values of the PS 80 samples with OA spiking.

% spiked OA in PS 80	**I_A**
0.00%	1.5
0.81%	3.1
1.98%	5.4
3.01%	7.5
3.98%	9.4
5.02%	11.4