

Supporting Information

Monitoring of the Degree of Condensation in Alkoxysiloxane Layers by NIR Reflection Spectroscopy

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Table S1. Ratio of T¹, T², and T³ determined by ²⁹Si NMR spectroscopy

Batch	Reaction Time [min]	Batch Size [mol]	Catalyst / mol MEMO	T ¹ [%]	T ² [%]	T ³ [%]
1	10	1	3 mmol HCl	4	53	43
2	15	1	3 mmol HCl	6	53	41
3	30	1	3 mmol HCl	3	60	37
4	45	1	3 mmol HCl	3	56	41
5	60	1	3 mmol HCl	2	55	43
6	120	1	3 mmol HCl	2	56	42
7	1320	1	3 mmol HCl	2	40	58
8	15	0.1	3 mmol HCl	4	57	39
9	30	0.1	3 mmol HCl	4	58	38
10	60	0.1	3 mmol HCl	3	60	37
11	120	0.1	3 mmol HCl	5	55	40
12	300	0,1	3 mmol HCl	3	49	48
13	120	0.1	3 mmol HCl	2	44	54
14	1440	0.1	3 mmol HCl	5	42	53
15	10	0.1	3 mmol HCl	9	64	27
16	45	0.1	3 mmol HCl	5	66	29
17	120	0.1	3 mmol HCl	4	59	37
18 [#]	88	0.1	3 mmol AceticAcid	48	36	3
19 [#]	240	0.1	30 mmol Acetic Acid	46	47	6
20 [#]	90	0.1	3 mmol Acetic Acid / 0.3 mmol HCl	2	52	8
21 [#]	120	0.1	0.3 mmol Acetic Acid	34	37	6
22 [#]	240	0.1	0.3 mmol Acetic Acid	21	42	30

[#] missing percent to 100 % correspond to hydrolyzed monomers T⁰

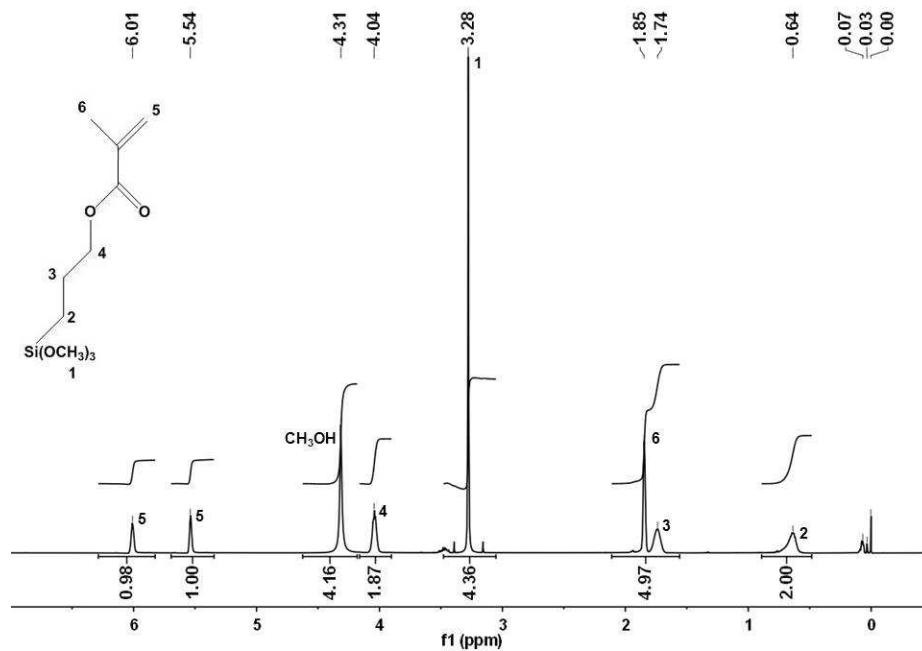


Figure S1: ^1H NMR spectrum of a typical sample (batch 15)

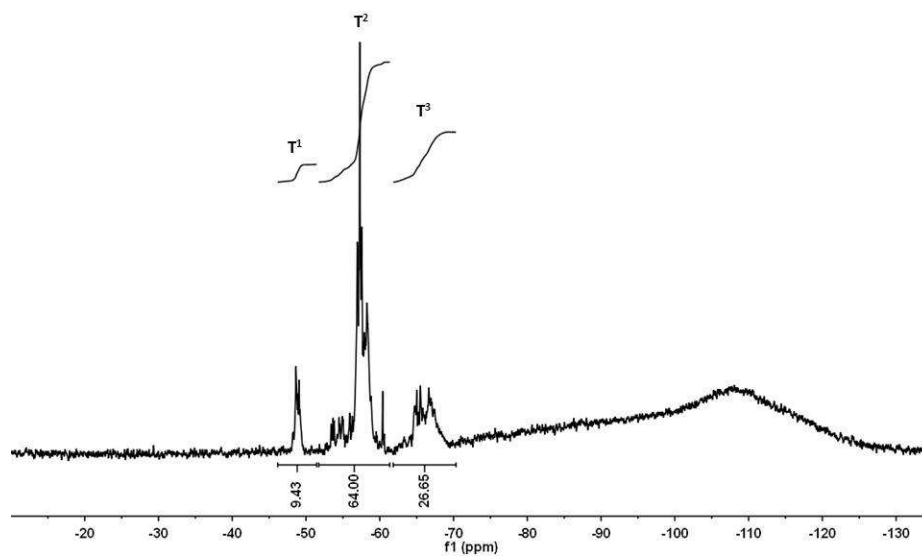


Figure S2: ^{29}Si NMR spectrum of a typical sample (batch 15) as used for calibration and validation

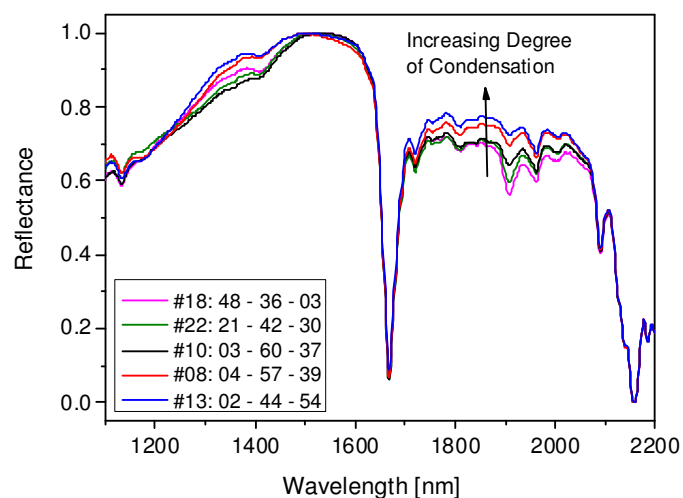


Figure S3a: Effect of an increasing degree of condensation of siloxane layers on PEN film on the NIR spectrum. The legend gives the number of the sample and the corresponding percentages of T^1 , T^2 , and T^3 (see Tab. S-1).

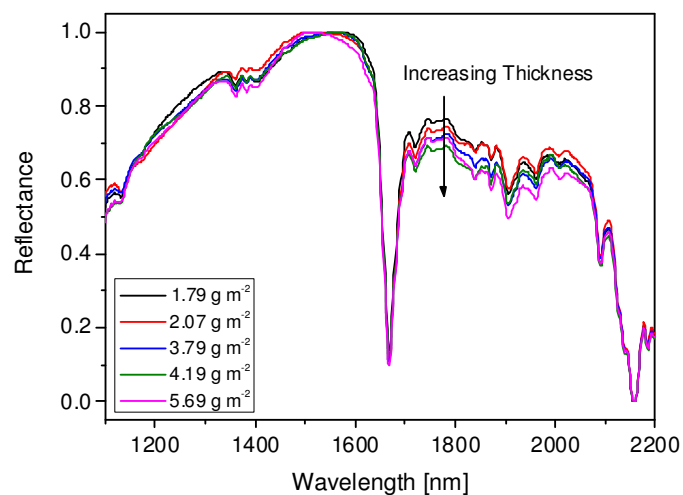


Figure S3b: Effect of increasing thickness of partially condensed siloxane layers on PEN film on the NIR spectrum.

Calibration and prediction plots shown in the printed paper

In order to give the reader a sense for the distance of the data to the unit slope line in all calibration and prediction plots shown in the printed paper, we added additional lines for $\pm 10\%$ relative deviation, that is lines with slopes 0.9 and 1.1, respectively, and an intercept at zero. Whereas the figures in the printed paper are given without these lines for reasons of clarity, the following pages will show the advanced plots.

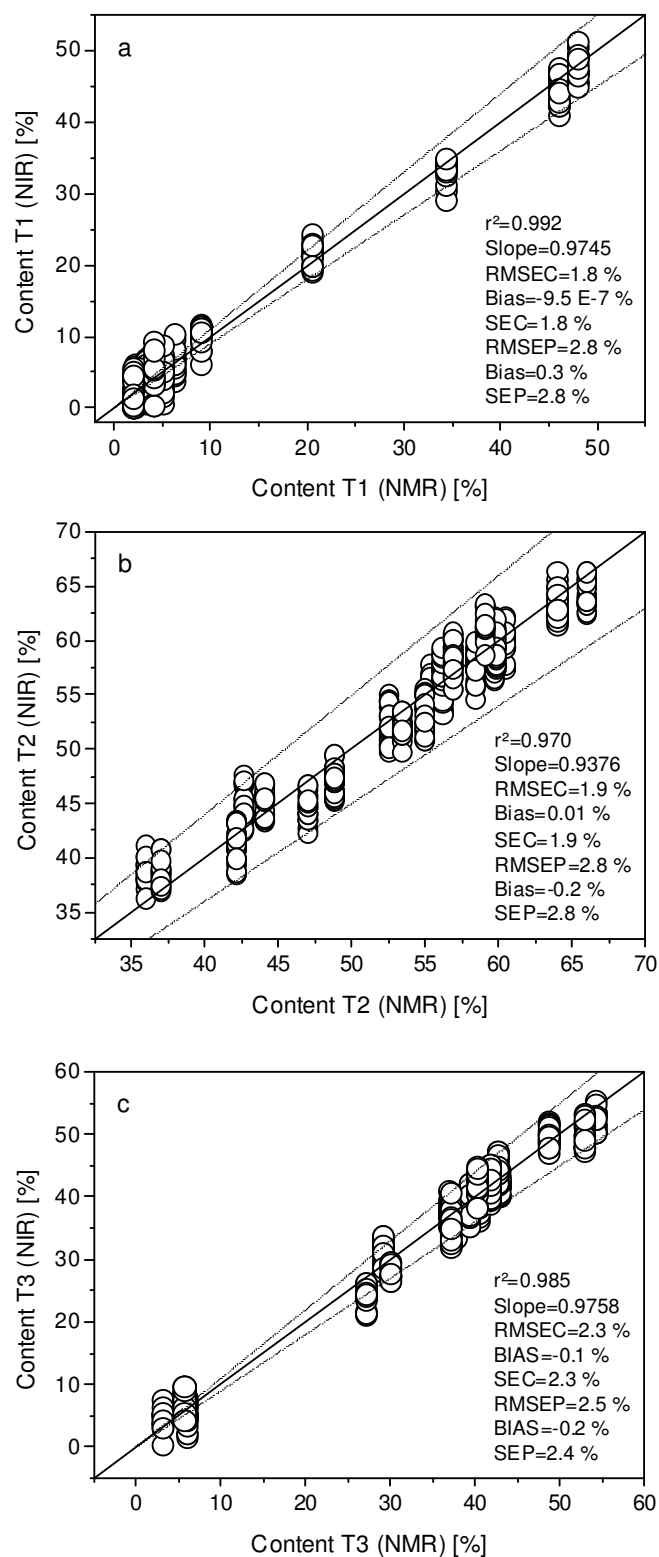


Fig. S4: PLS2 calibration model for siloxanes: 2D projections of the calibration data to the content of T^1 (a), T^2 (b), and T^3 (c) species, respectively (modified version of Fig. 1).

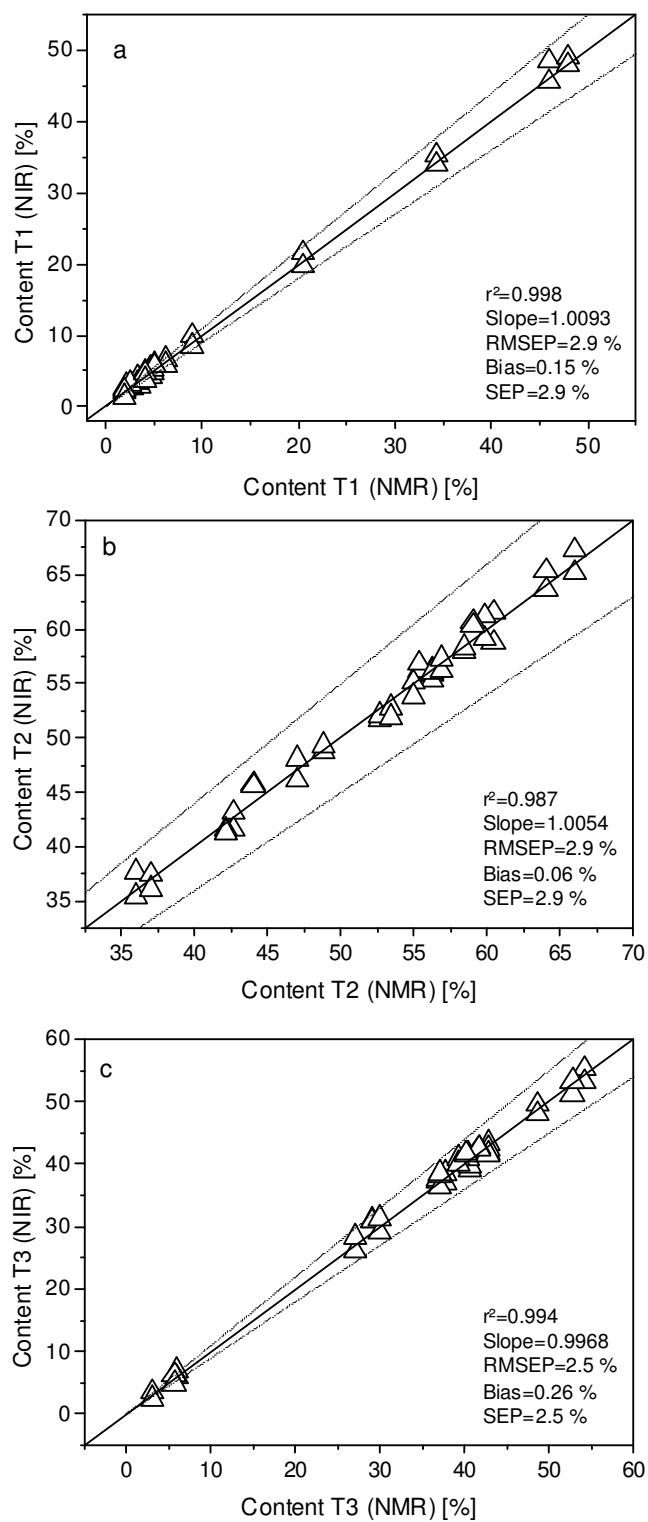


Fig. S5: Prediction of the degree of condensation in independent samples of the siloxanes using the PLS2 calibration model shown in Fig. 1 (modified version of Fig. 2).

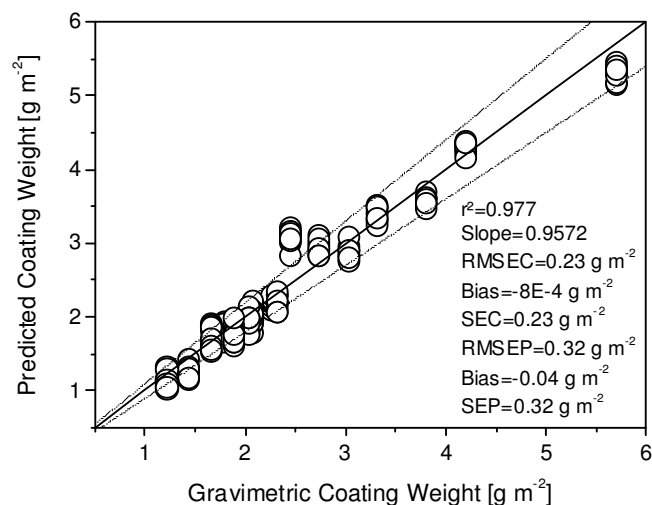


Fig. S6: PLS1 calibration model for the coating weight of thin siloxane layers on PEN film (modified version of Fig. 3).

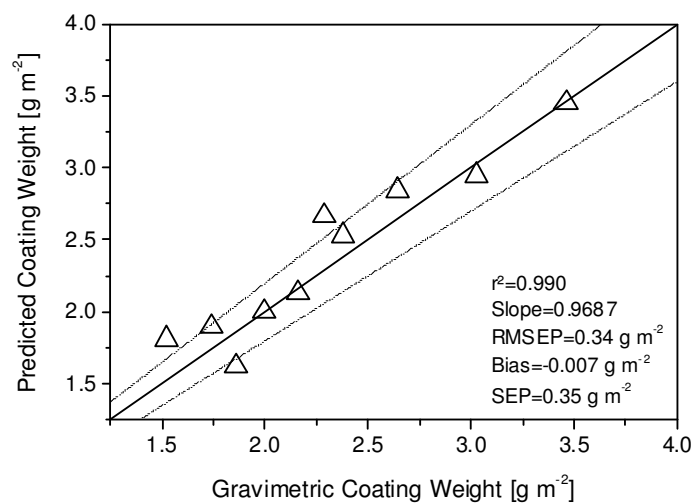


Fig. S7: Prediction of the coating weights of independent samples of siloxane layers on PEN film (modified version of Fig. 4).