

# Sorbents for Removal Surfactants from Aqueous Solutions: Surface Modification of Natural Solids to Enhance Sorption Ability

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**ABSTRACT:** Particulate materials of natural origin, sulfite cellulose and two kaolinite clays, were modified by the formation of nonstoichiometric polyelectrolyte complex on the surface of particles. This resulted in positive surface charge on cellulose and clays. Modified materials as well as selected commercial polymers were used as sorbents for removal of anionic, nonionic, and cationic surfactants from aqueous solutions. It was stated that cationic surface modi-

fication of cellulose and kaolinite clays resulted in a significant increase of sorption rate and degree of removal of oppositely charged anionic surfactants. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 92: 1510–1515, 2004

**Key words:** adsorption; clay; micelles; polyelectrolytes; surfactants

## INTRODUCTION

Sorption of surfactants from some kinds of industrial wastewater by polymeric or mineral sorbents may effectively reduce their concentration and undesired influence on the environment. Sorption is a result of complex interaction between a surfactant and a sorbent, including electrostatic and hydrophobic interactions,<sup>1–5</sup> formation of hydrogen bonds, etc. Hydrogel/ionic surfactant interactions are also governed by translation entropy of counterions, because the surfactant ions penetrate in the network and replace the network counterions. Electrostatic binding is a process driven by its positive entropy attributed to the recovery of translation entropy of counterions released by the bound surfactant.<sup>6</sup> If the volume charge density in the network is much higher than in the external solution, the surfactant ions are effectively concentrated within the network.<sup>2,7</sup> Because of the hydrophobic interaction between their chains, surfactant ions aggregate within polymer network.<sup>8</sup> The aggregation of an ionic surfactant starts at a concentration that is much lower than the critical micellar concentration (CMC) of the surfactant in water.<sup>2,9</sup> This aggregation is

responsible for the decrease in the concentration of mobile counterions in the network and thus for the decrease of internal osmotic pressure in the hydrogel and to the collapse of the hydrogel. After the volume collapse of an oppositely charged gel, enhanced velocity of surfactant binding was observed because of increased charge density, despite the decreased free volume for diffusion.<sup>10</sup>

It could be expected that the sorption mechanism would be simpler and the process would be faster when the charge would be located on the surface of sorbent particles. Such particles could be produced by surface modification of inert particles, which would generate surface charge. From a practical point of view, such hybrid sorbents would be interesting, when both initial particles would be cheap (such as some materials of natural origin); the modification process would be easy and, of course, sorption properties would be acceptable.

Commercial surfactants are mainly of anionic character, although cationic and nonionic ones are also produced in considerable amounts. For this reason, the sorbents with basic properties, providing them the ability to absorb anionic surfactants because of electrostatic interactions,<sup>4,5</sup> seemed to be the most important. A typical anionic surfactant, such as dodecylbenzene sulfonate, is removed from wastewater effluence by biodegradation, which is slow and takes up to 15 days<sup>11</sup> or adsorption onto mineral adsorbents. Kaolinite belongs to the most common adsorbents.<sup>12,13</sup>

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Hanna and Somasundaran assigned the sodium dodecylbenzenesulfonate (SDBS) adsorption onto the kaolinite surface to a combination of mechanisms involving electrostatic adsorption, ionic exchange, adsorption activated by metals, and precipitation.<sup>14</sup> Recently, it was found that subsequent deposition of cationic and anionic polyelectrolytes on the particles of sulfite cellulose or kaolinite clay may be an easy and effective method of cationic modification of their surface.<sup>15,16,17</sup> It could be expected that creation of positive charge on the surface of cellulose fibers or a clay particle would enhance the sorption of SDBS and other anionic surfactants because of electrostatic effect.

The aim of this work was to estimate the influence of cationic modification of cellulose and clay particles on their sorption properties toward typical industrial surfactants and to compare them with selected commercial products as reference materials.

## EXPERIMENTAL

### Materials

Cellulose, beaten pure spruce sulfite cellulose with a degree of milling equal to 34°SR (i.e., with medium specific surface area), was used in sorption measurements in air-dried state, but modified with polyelectrolyte complex in wet state. Chitosan (Acros Organics), Amberlite IRA410 (Fluka, Buchs, Switzerland) ( $d = 1.12$  g/mL, moisture content, 42%, 0.48 mm), as well as two kaolinite clays, Frechen and TECFF 840 (Quarwerke, Frechen, Germany and Amberger Kaolinwerke, Amberg, Germany), were used as received in all experiments.

Hydrogel sorbent based on neutralized, crosslinked poly(acrylic acid), Favor SXM 9130 (Stockausen, Degusa, kindly provided by Hartmann AG, Germany), was used in dry state as received.

Technical-grade surfactants of sodium dodecylbenzenesulfonate (paste) and oxyethylenated *n*-nonylphenol Rokafenol N-8 were delivered by Z. Ch Rokita Co. (Brzey, Poland). The former was dried before the use to get anhydrous powder, and *N*-dodecylpyridinium bromide and *N*-dodecylpyridinium chloride (Merck, Darmstadt, Germany) were used as received.

### Estimation of CMC

CMC values were determined graphically from dependence of surface tension on the logarithm of the concentration of a surfactant. Surface tension (0.0319–0.0709 N/m) was measured by stalagmometric method for  $7.5 \times 10^{-6}$ – $2.41 \times 10^{-2}$  mol/dm<sup>3</sup> solutions of the surfactants in distilled water at room temperature. Stalagmometer was calibrated with distilled water and surface tension was calculated according to (1):

$$D_x = D_w N_w / N_x \quad (\text{N/m}) \quad (1)$$

where  $D_w$  is the surface tension of water, and  $D_w = 0.07253$  N/m at 20°C<sup>18</sup>;  $N_w$  and  $N_x$  are the amount of droplets of water and amount of the solution formed from the volume of a liquid contained in the stalagmometer.

### Surface modification by deposition of polyelectrolytes

The surface modification of cellulose and clays by polyelectrolyte deposition, poly(diallyldimethylammonium chloride) (PDADMAC) and sodium salt of poly(maleic acid-co- $\alpha$ -methylstyrene) (PMAMS), was carried out as described elsewhere.<sup>15–17</sup> Briefly, 1 g of dry material was suspended in distilled water, 30 mL of 0.01M solution of PDADMAC was added under continuous stirring, and then after 2 min, polyanion was added as 0.01M solution in such an amount that  $n^-/n^+$  was 0.55–0.6 for cellulose or 0.6–0.7 for clays and the volume of suspension was 100 cm<sup>3</sup>. The pH was adjusted to 7; the modified particles were separated, washed with water, and air-dried. In the case of clay TEC FF 840, the modified material was spray dried.

### Characterization of the sorbents and their sorption properties

Particle size of the clays was measured by using HELOS particle-size analyzer (Sympatec GmbH, Germany). Surface charge of the sorbents was determined by polyelectrolyte titration by using particle charge detector PCD 02 (Mütek GmbH, Germany) in combination with a Mettler Titrator DL 21 as described elsewhere.<sup>16</sup>

Swelling properties [water sorption, WS (g H<sub>2</sub>O/g) and equilibrium water content, EWC (%)] were determined gravimetrically after 7 days soaking of a sample of a hydrogel in an excess of distilled water.<sup>19</sup>

The sorption of SDBS and ethoxylated phenol Rokafenol N-8 (RF-8), as well as that of *N*-dodecyl pyridinium bromide (NDPBr) or chloride (NDPCL) from diluted aqueous solutions (0.2, 0.4, 1.0, and 2.0 g/dm<sup>3</sup>), was characterized by determination of their concentration after a predetermined period (1 h, 1 day, and 7 days) of contacting the surfactant solution (50 cm<sup>3</sup>) with a sorbent (0.1 g). Concentration of a surfactant in a solution was calculated from absorbency at  $\lambda_{\text{max}}$ , which was determined with UV-2102 PC spectrometer (UV-VIS Scanning Spectrophotometer, Shimadzu) coupled with a computer. SDBS, RF-8, NDPBr, or NDPCL display strong UV absorption at 224, 223, and 259 nm, respectively.

## RESULTS AND DISCUSSION

As shown previously,<sup>15–17</sup> the interaction between two oppositely charged polyelectrolytes in the presence of a solid can be used as a simple way for cationic modification of the surface of particles. Positive charge at the surface of the solids would support the sorption of anionic surfactants from their aqueous solutions because of the electrostatic interaction between the surface and a surfactant. In this work, sulfite cellulose and two kinds of clays were modified according to a known procedure.<sup>15–17</sup> That procedure comprises a two-step formation of nonstoichiometric polyelectrolyte complex. The first polycation (PDADMAC) is deposited on particles with small negative surface charge (cellulose or clay particles); then particles are contacted with the solution of polyanion [sodium salt of poly(maleic acid-*co*- $\alpha$ -methylstyrene)]. A strong enhancement of specific cationic surface charge was observed at a ratio of anionic-to-cationic charges  $n^-/n^+$  equal to 0.6–0.7 (clays) or 0.55–0.60 (cellulose), but results were strongly affected by the conditions of modification, especially by the concentration of solid particles.

All the products obtained as well as selected commercial particulate materials of cationic (Amberlite IRA 410), cationogenic (chitosan), and anionic (Favor SXM 9130) character, considered here as sorbents, were checked for their sorption properties toward anionic surfactant–sodium dodecylbenzenesulfate. The sorption of nonionic- and cationic-type surfactants was measured as well to estimate the role of other mechanisms of the sorption.

### Estimation of CMC

It is known that sorption of the surfactants may depend on their concentration because of the formation of micelles in the solution and aggregates within the swelled polymer network.<sup>6,20</sup> Micellar solutions can be also more important from a practical point of view. For this reason, it seemed important to know if the surfactant studied existed in the form of micelles in the solution used in sorption measurements. CMC values obtained from stalagmometric measurements were in the range of 0.40–1.86 g/dm<sup>3</sup> (Table I), whereas the concentration of the solutions in sorption experiments were 0.2–2.0 g/dm<sup>3</sup>. It means that, in the most diluted solutions (0.2 g/dm<sup>3</sup>), there is no micellization, but, in more concentrated solutions, a part of the surfactant may exist in the form of micelles. The critical aggregation concentrations of a surfactant within polymer network are usually much lower than CMC values,<sup>2,9</sup> so for all the solutions studied, the concentration of the surfactants is high enough to make possible thermodynamic mechanism when hydrogel sorbent Favor SXM 9130 and probably also chitosan are used.

TABLE I  
Critical Micellar Concentration of the Surfactants Used in Sorption Experiments Obtained from Stalagmometric Measurements at 20°C

Surfactant	CMC values measured		Lit. CMC value [mmol/dm <sup>3</sup> ]
	[mmol/ dm <sup>3</sup> ]	[g/ dm <sup>3</sup> ]	
SDBS	1.14	0.40	0.63 (25°C) <sup>21</sup> 1.59 (30°C) <sup>13</sup>
RF-8	1.41	0.81	—
NDPBr	5.66	1.86	—
NDPCI	5.55	1.56	—

### Sorption of surfactants

Degree of surfactant removal from aqueous solutions was measured after a short contact time with a sorbent as well as after a long period of contacting a sorbent with a solution of a surfactant. It was assumed that a short contact sorption time may give information about effectiveness of a sorbent in the fast removal of a surfactant, whereas a long observation time would give information about the sorption capacity.

### Short sorption time

Results of the sorption measurement after short contact time of diluted solutions with a sorbent are collected in Tables II and III.

It can be seen that despite the cationic character of the surface of modified cellulose, the sorption of anionic SDBS, as well as that of other surfactants after 1 h, is very weak. Degree of SDBS removal from aqueous solution is even lower than that observed for unmodified cellulose. It suggested that the mechanism of electrostatic interaction did not work here, probably because of hindered swelling and diffusion of water and of SDBS to microfibrils inside of modified cellulose fibers. Only surface sorption could be expected in the case of clay particles. Two kaolinite clays studied display considerable sorption of cationic surfactant, probably because of small negative surface charge. Their modification by two-step deposition of polyelectrolytes resulted in agglomeration of particles and appeared very effective in terms of the creation of positive surface charge as well as in terms of increased sorption of anionic surfactant. The best sorption properties toward SDBS was observed for surface-modified clay TECFF 840, probably because of a bigger specific surface of initial particles than that of clay Frechen. Within a relatively short contact time, modified clay TECFF 840 is capable of removing ~ 70% of SDBS from its diluted aqueous solution, so the sorption effectiveness is ~ 10 times higher than that of unmodified clay.

Relatively low sorption activity displays aminofunctional polysaccharide–chitosan and strong anion exchange resin Amberlite IRA 410.

TABLE II  
Degree of Surfactant Removal from Aqueous Solution (0.2g/dm<sup>3</sup>) after 1 h Contacting with a Sorbent

Sorbent	Modifier	$D_n$ ( $\mu\text{m}$ )	Surface charge ( $\mu\text{mol/g}$ )	WS (g/g)	SDBS sorption (%)	RF-8 sorption (%)	NDPBr sorption (%)
Cellulose	—	—	2.5 (-)	2.07	14.3	1.6	0.0
Mod. cellulose (Mo1)	PDADMAC/PMAMS	—	13.6 (+)	—	7.2	0.0	0.65
Mod. cellulose (Mo2)	PDADMAC/PMAMS	—	12.8 (+)	—	8.5	0.0	0.65
Clay frechen	—	15.8	1.18 (-)	—	8.2	0.0	28.1 <sup>a</sup>
Modified clay frechen	PDADMAC/PMAMS	>50	49.0 (+)	—	36.5	0.76	1.8 <sup>a</sup>
Clay TECFF 840	—	7.3	6.0 (-)	—	6.9	0.0	30.0 <sup>a</sup>
Modified clay TECFF 840	PDADMAC/PMAMS	38.8	26.1 (+)	—	70.8	0.22	1.6 <sup>a</sup>
Chitosan	—	—	—	4.03	30.6	0.0	0.0
Amberlite IRA-410	—	480	1650 (+) <sup>b</sup>	0.81	17.3	5.7	1.8
Favor SXM9130	—	—	—	280	5.7	2.3	82.2

<sup>a</sup> N-dodecylpyridinium chloride used instead of N-dodecylpyridinium bromide.

<sup>b</sup> Estimated from exchange capacity and apparent density of wet resin.<sup>22</sup>

According to our expectations, anionic hydrogel sorbent Favor displays only low-sorption activity toward SDBS, but it was a very effective sorbent for cationic surfactant NDPBr. It suggested that electrostatic interaction dominates over other mechanism of the sorption expected in this case (i.e., oversorption because of differences in free energy of micelles in the solution and in the polymer network).

### Long sorption time

Sorption of the surfactants was observed during long contact time of their solutions (up to 7 days) with the sorbents. For more effective sorbents, measurements were carried out also by using more concentrated solutions of selected ionic surfactants to get information about both time profile of the sorption as well as sorption capacity. It appeared that the time profile of the sorption depended very much on the nature of the sorbent.

Degree of cationic surfactant removal by anionic hydrogel sorbent Favor SXM 9130 increased only

moderately with time of the contact, especially when more concentrated surfactant solutions were used. It is a result of very fast swelling of the material and easy penetration of the surfactant molecules into swelled hydrogel particles. The material is swelled almost completely already after 1 h (WS = 260 g/g, while in equilibrium WS = 280 g/g) and after 1 day sorption, the degree of surfactant removal remains practically unchanged.

Chitosan particles swell moderately (WS = 4.03 g H<sub>2</sub>O/g, EWC = 80.1%) and only a small increase of surfactant removal was observed after a longer time of exposition.

Polymer network of typical gel-type ion exchange resin Amberlite IRA 410 is rather dense and swelling in water is limited (EWC = 44.8%). According to information from Fluka,<sup>22</sup> apparent porosity of such type of resin usually does not exceed 40 Å, and therefore, penetration of a surfactant into a particle is slow. It is reflected in a distinct increase of degree of surfactant removal with time of sorption up to 7 days.

TABLE III  
Degree of Surfactant Removal from Aqueous Solution (0.4g/dm<sup>3</sup>) after 1 h Contacting with a Sorbent

Sorbent	Modifier	$D_a$ ( $\mu\text{m}$ )	Surface charge ( $\mu\text{mol/g}$ )	SDBS sorption (%)	RF-8 sorption (%)	NDPBr sorption (%)
Cellulose	—	—	2.5 (-)	14.8	10.1	12.3
Cellulose (Mo1)	PDADMAC/PMAMS	—	13.6 (+)	7.3	5.6	11.0
Cellulose (Mo2)	PDADMAC/PMAMS	—	12.8 (+)	5.4	5.8	8.8
Clay frechen	—	15.8	1.18(-)	9.4	6.4	42.2 <sup>a</sup>
Mod. clay frechen	PDADMAC/PMAMS	>50	49.0 (+)	39.6	0.4	0.0 <sup>a</sup>
Clay TECFF 840	—	7.3	6.0 (-)	—	—	—
Mod. clay TECFF 840	PDADMAC/PMAMS	38.8	26.1 (+)	—	—	—
Chitosan	—	—	—	23.8	0	4.1
Amberlite IRA-410	—	480	—	20.4	9.6	7.4
Favor SXM 9130	—	—	—	15.1	18.0	88.0

<sup>a</sup> N-dodecylpyridinium chloride instead of bromide.



TABLE IV  
Selected Results of SDBS Sorption from its Aqueous Solutions after Various Period of Contacting with a Sorbent

Sorbent	Modifier	Surfactant concentration (g/l)	Degree of surfactant removal (%) after contact time with a sorbent			Sorption capacity (gSDBS/1 g) after 168 h
			1 h	24 h	168 h	
Cellulose	—	0.2	14.3	14.9	15.2	0.015
Mod. cellulose (Mol)	PDADMAC/PMAMS	0.2	7.2	37.8	62.0	0.062
		0.4	7.3	54.0	73.4	0.146
		1.0	3.8	18.8	35.9	0.180
		2.0	9.4	24.4	42.5	0.425
Mod. cellulose (Mo2)	PDADMAC/PMAMS	0.2	8.5	34.7	54.5	0.055
		0.4	5.4	52.2	77.7	0.154
		1.0	5.9	15.3	36.6	0.183
		2.0	10.8	19.0	32.9	0.329
Clay frechen	—	0.2	8.2	9.8	10.1	0.010
Modified clay frechen	PDADMAC/PMAMS	0.2	36.5	65.6	70.2	0.070
		0.4	39.6	74.1	77.8	0.156
Clay TECFF 840	—	0.2	6.9	6.9	7.2	0.010
Modified clay TECFF 840	PDADMAC/PMAMS	0.2	70.8	73.5	73.9	0.074
		0.4	30.6	30.8	31.0	0.031
Chitosan	—	0.2	23.8	50.1	50.2	0.100
		0.4	17.3	53.1	78.3	0.078
		0.4	20.4	77.1	91.1	0.182
		1.0	19.0	75.3	90.1	0.455
Amberlite IRA-410	—	1.0	15.2	70.2	84.9	0.849
		2.0	15.2	70.2	84.9	0.849

With respect to the aim of this work, the most interesting finding was, however, the estimation of the results of cationic modification of selected natural solids and comparison of their sorption properties toward anionic surfactants with those of reference materials. Results of the sorption measurements are gathered in Table IV.

The degree of surfactant removal by unmodified sorbents, cellulose and kaolinite clays, does not change practically during the observation and remains less or more at the same low level as found after 1 h contact with a surfactant solution. It is probably due to the surface character of the sorption process.

Modification of cellulose and clays led to a change of time profile of the sorption. In all cases, one may observe an increasing degree of surfactant removal with elongation of the process, especially when more concentrated solutions of the surfactants were used. Time dependence of the sorption is surprising, especially in the case of modified clays, because only surface adsorption of oppositely charged surfactants was expected. Both modified clays, Frechen and TECFF, appeared to be quite effective sorbents for anionic surfactant SDBS, because they are able to remove 70–80% of the surfactant, whereas before modification, degree of removal was only 7–10%.

Due to the ionic character of the sorbents mentioned in Table IV (excepting chitosan) and of the SDBS, one may assume electrostatic interaction as main mechanism of the sorption. No stoichiometry was found, however, when degrees of surfactant removal from

aqueous solutions of various concentrations were compared. Calculation of the relation between amount of micromoles of SDBS bound to 1 g of a sorbent after 7 days and amount of micromoles of positive charges measured for 1 g of the sorbent [SDBS/(positive charge of 1g of a sorbent), micromol/micromol] revealed that only in the case of Amberlite IRA 410 that the relation is not far away from the value expected from the electrostatic mechanism (i.e., 1:1). It depended very much on the concentration of SDBS solution and is equal to only 0.14 for the most diluted solution (0.2 g/L) but increased up to 1.48 for the sorption from the most concentrated (2.0 g/L) solution. For that calculation, total exchange capacity of the resin was considered as the positive charge of 1 g Amberlite. The same tendency (i.e., increase of the relation with increased SDBS concentration) was observed for modified cellulose and clays, but the values of that relation were much higher—up to 9.1 for the clays and up to ~ 90 for modified cellulose. It means that the process is more complex than simple electrostatic interaction between oppositely charged sorbent and surfactant.

The sorption capacity (i.e., amount of SDBS removed by 1 g of the sorbent after 7 days) depended also on the concentration of SDBS solution and increased with the concentration of the latter. Despite the fact that, in modified cellulose and clays, only particle surfaces cumulate positive charge, their sorption capacity calculated from degree of surfactant removal after 7 days (~ 0.06 g SDBS/1 g) is similar to

that of Amberlite ( $\sim 0.08$  g SDBS/1 g). It is, however, lower for more concentrated SDBS solution ( $\sim 0.3$ – $0.4$  for modified cellulose and  $0.85$  g SDBS/1 g for Amberlite).

### CONCLUSION

Two-step deposition of oppositely charged polyelectrolytes on the surface of cellulose or kaolinite clays appeared to be a convenient method of cationic modification of particulate or fibrous solids, providing them an ability to remove an anionic surfactant, (i.e., sodium dodecylbenzenesulfonate) from aqueous solutions.

Degree of anionic surfactant removal by modified clays or cellulose, as well as their sorption capacity, is up to one order higher than those of initial unmodified solids and comparable with that of gel type anionic exchange resin Amberlite IRA 410.

Due to mainly surface character, the sorption of anionic surfactant on modified clays, especially on modified clay TECFF 840 (with larger surface, when comparing with clay Frechen, due to spray drying), is faster than diffusion-controlled sorption by ion exchange resin.

Amount of anionic surfactant bound onto/in the sorbent increases with elongation of the sorption time and increasing concentration of the surfactant. Time dependence of the amount of bound surfactant is most significant for ion exchange resin and much less for modified clays.

Amount of anionic surfactant bound onto modified cellulose or clays is much higher than that expected from their surface charge. This suggested that the mechanism of the sorption is more complex than simple electrostatic interaction between anionic surfactant and cationic surface, which probably only initiates the sorption process.

Relatively weak sorption ability of nonionic surfactant does not change and sorption of cationic surfactants decreases as a result of the modification.

Removal of oppositely charged (i.e., anionic) surfactant is relatively fast because of mainly surface character of the sorption process and minimal influence of the diffusion to the particle interior.

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