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ORIGINAL ARTICLE

Synthesis, Characterization and Surface Activity of New Eco-friendly Schiff Bases Vanillin Derived Cationic Surfactants

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Abstract Three eco-friendly cationic surface active agents were synthesized from the chemical modification of vanillin. The chemical structures of these surfactants were confirmed using elemental analysis, IR and NMR spectra. The surface activity measurements showed their high tendency towards adsorption and micellization and their good surface tension reduction, low interfacial tension. The emulsion stability measurements showed acceptable efficiency as emulsifying agents for short term emulsions. The biodegradability tests revealed that these compounds are eco-friendly and had completely degraded in 30 days.

Keywords Vanillin · Eco-friendly · Surface activity · Schiff base · Biodegradation

Introduction

Natural occurring compounds are those compounds which occur in different organisms, animals, plants or microorganisms. These compounds are different and widely spread in the plant and animal kingdoms. One of the most widespread compounds in the plant kingdom is vanillin. Vanillin is white or lightly yellow solid extracted from the vanilla bean. It is also found in roasted coffee and the Chinese red pine. Several studies were done on the vanillin and its application as pesticide, green corrosion inhibitors for corrosion of different metals. The urge of modification of natural compounds in different applications is due to the continuous accumulation of the chemical compounds in the environment and the difficulty of their degradation. Then, eco-friendly compounds were the solution of the environmental pollution [1–6]. In this study, naturally occurring vanillin was chemically modified into cationic surface active agents containing hydrocarbon chains with different lengths. The surface activity and some surface properties of the produced cationic vanillin derivatives were determined. In addition, the biodegradability of the new synthesized surfactants was measured.

Experimental

Chemicals

Vanillin (4-Hydroxy-3-methoxybenzaldehyde), 3-aminopyridine, bromoacetic acid and fatty alcohols were purchased from Aldrich, Germany.

Synthesis

Synthesis of Alkyl Bromoacetate (OB, DB, HB)

Bromoacetic acid (0.1 mol) and octyl, dodecyl and hexadecyl alcohol (0.1 mol) were esterified, respectively in xylene in the presence of 0.1% *p*-toluene sulfonic acid (a catalyst), until the azeotropic amount of water (1.8 mL) was removed. After removal of the solvent under vacuum using a rotary evaporator, the catalyst was extracted from

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the reaction medium using petroleum ether. Subsequent purification was done by extracting the unreacted material with petroleum ether [7] to afford the different fatty alcohol esters of bromoacetic acid which are designated as octyl bromoacetate (OB), dodecyl bromoacetate (DB) and hexadecyl bromoacetate (HB).

Compound LB: IR: 2,940 cm^{-1} (CH_3), 2,850 cm^{-1} (CH_2), 1,736 cm^{-1} (C=O), 554 cm^{-1} (C–Br). ^{1}H NMR ($CDCl_3$): 0.9 ppm (t, 3H, $–CH_3$), 1.36 ppm (m, 20H, $–CH_2–$), 3.35 ppm ($–O–CH_2–$), 4.17 ppm ($Br–CH_2–$).

Synthesis of 3-Aminopyridine-Vanillin Schiff Base (VSB)

Vanillin and 3-amino pyridine were mixed together in equimolar ratio in a 250-mL round-bottom flask in the presence of 50 mL ethanol as a solvent and 0.1% *p*-toluene sulfonic acid as a dehydrating agent. The mixture was refluxed under stirring for 4 h and then left to precipitate. The product was filtered off and recrystallized twice from ethanol to obtain the desired vanillin Schiff base (VSB) [8].

VSB: IR: 3,400 cm^{-1} (O–H), 2,850 cm^{-1} (CH_3), 1,628 cm^{-1} (–C=N–, azomethine), 1,051 cm^{-1} ($O–CH_3$), 870 cm^{-1} (aromatic protons of phenyl group). ^{1}H NMR ($CDCl_3$): 3.73 ppm (s, 3H, $–OCH_3$), 7.26 ppm (t, 2H, –CH=CH– benzylidenimine), 8.1 ppm (s, 1H, –N = CH– azomethine).

Synthesis of the Cationic Schiff Base Derivatives of Vanillin (VSBO, VSBD, VSBH)

Equimolar amounts of the vanillin Schiff base (*VSB*) and alkyl bromoacetate esters (*OB*, *DB*, *HB*) were refluxed individually in acetone (100 mL) as a solvent for 4 h and then left to cool. The products were filtered off and recrystallized twice from ethanol to remove the unreactants and the residuals, and then dried under a vacuum at 50 °C for 6 h to yield the desired cationic quaternary compounds which were designated as *VSBO* for octyl derivative, *VSBD* for dodecyl derivative and *VSBH* for hexadecyl derivative [9].

Compound *VSBD*. IR: 1,628 cm^{-1} (–C=N–, azomethine), 870 cm^{-1} (aromatic protons of phenyl group), 2,920 cm^{-1} (CH_2), 2,850 cm^{-1} (CH_3), 3,040, 1,490 cm^{-1} (N^+), 1,051 cm^{-1} ($O–CH_3$). ^{1}H NMR ($CDCl_3$): 0.96 ppm (t, 3H, $–CH_3$), 1.3 ppm (m, 20H, $–CH_2–[CH_2]_{10}–CH_3$), 3.73 ppm (s, 3H, $–OCH_3$), 4.19 ppm (t, 2H, $–COO–CH_2$), 7.26 ppm (t, 2H, =CH–CH=benzylidenimine), 7.33 ppm, 8.1 ppm (s, 1H, –N=CH– azomethine). ^{13}C NMR ($CDCl_3$): 14.1 ppm ($–CH_3$), 22.8 ppm ($–CH_2CH_3$), 29.7 ppm ($CH_2[CH_2]_9CH_2CH_3$), 55.9 ppm ($–OCH_3$), 63.2 ppm (OCH_2), 118.3 ppm (=CH–CH=CH–CH=benzylidenimine), 160.2 ppm (–N = **C**H– azomethine), 161 ppm ($–COOCH_2$).

Measurements

Surface Tension (γ) and Interfacial Tension (γ_{IT}) Measurements

Freshly prepared aqueous cationic surfactants solutions with a concentration range of 0.01–0.000005 ML^{-1} were poured into a clean 25-mL Teflon holder and allowed to equilibrate for 2 h. Then, the platinum ring was adjusted at the air–water interface of the surfactant solution. The reading was recorded when the ring detached itself from the solution surface. The platinum ring was removed after each reading, washed with diluted HCl followed by distilled water. Apparent surface tension values were taken as the average of three replicates at 25.0 ± 0.5 °C [10].

Interfacial tension was measured between two layers of surfactant solution and light paraffin oil at 25.0 ± 0.5 °C. The surfactant solution (15 mL of 0.1% wt) was added firstly into the Teflon holder and the platinum ring was adjusted to touch the solution surface. Light paraffin (15 mL) oil was then added smoothly onto the surface and the system was left to equilibrate for 2 h. The readings were recorded at the point which the ring detached itself from the aqueous to the organic layer (paraffin oil). The ring was removed and washed with acetone followed by distilled water and the measurements repeated three times for each surfactant. The interfacial tension values were taken as the mean values of three readings [11].

Emulsification Power

A 20-mL amount of a different surfactant solution (0.1%) was individually placed in a 100-mL cylinder and then 20 mL of the paraffin oil was added. The cylinder was shaken vigorously for 10 min and then allowed to settle. The time required to separate 18 mL of pure surfactant solution was recorded and the experiment was repeated three times for each surfactant. The average separation time of the three experiments was taken as an indication of the emulsification power of each surfactant [11].

Biodegradation Test

The Modified Screening Test [12] described in the Organization for the Economic Cooperation and Development (OECD) Guidelines was applied to determine the biodegradation. A mixture of equal volumes of secondary effluent from a wastewater treatment plant and garden soil aqueous suspension was used as inoculums for the biodegradation test.

Results and Discussion

Chemical Structures

The chemical structures of the synthesized vanillin derived cationic surfactants and all of their intermediates were confirmed by elemental analysis, IR and NMR data provided in the synthesis section and illustrated in Scheme 1.

Surface Activity

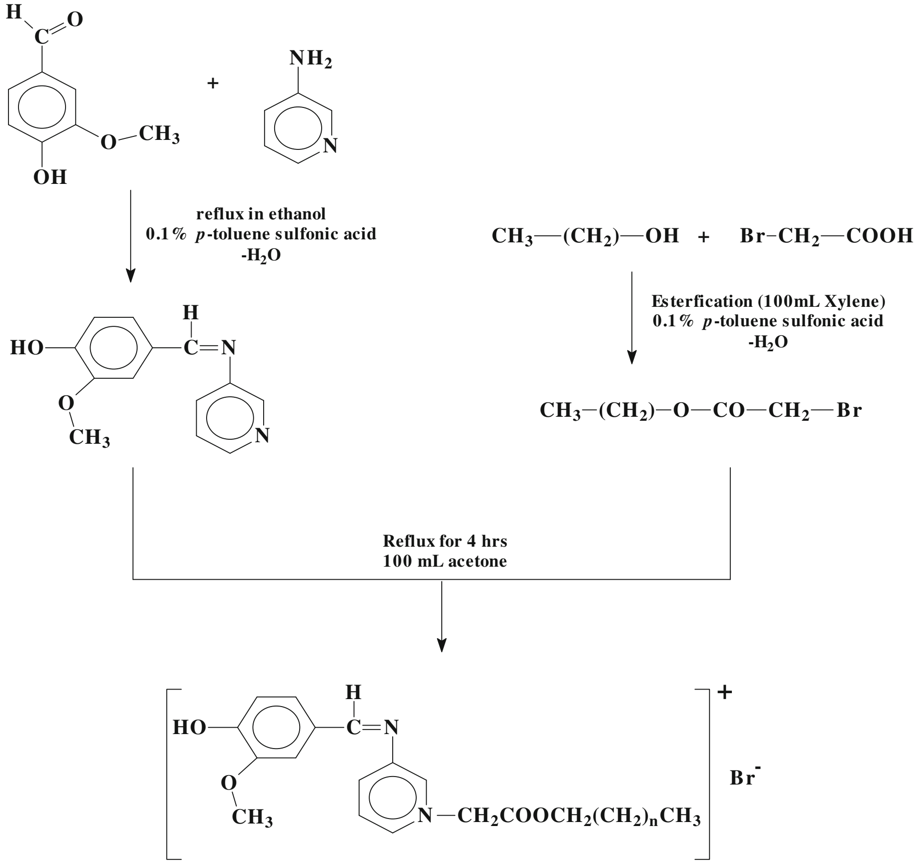
The hydrogen bonds formed between water molecules make the water surface curved. Contaminants associate with the water surface and break down the hydrogen bonds (depending on the quantity of these contaminants at the interface) and the surface tension of the water surface deviates from the absolute value of the pure water (71.8 mNm^{-1}) [13].

The amphipathic character of surfactant molecules is due to the presence of two characteristic parts: the hydrophobic (organic) and the hydrophilic part (polar). When surfactant molecules are dissolved in the aqueous medium, they organize in the solution in which the hydrophile is directed to the water phase. While, the hydrophobe is located on the interface to reduce the repulsion generated from the water phase. Increasing the amount of surfactants added to the solution decreases the surface tension values of the water gradually. The decrease in the surface tension values indicates the tendency of surfactant molecules towards adsorption at the liquid interface. At a certain concentration, surfactant molecules cover the water interface completely. In this case, surfactant molecules are aggregated in the bulk of the solution to form the micelles [14].

The surface tension versus −log concentration profiles of the vanillin derived cationic surfactant solutions *(VSBO, VSBD, VSBH)* showed two characteristic regions (Fig. 1). The first at low concentrations and characterized by continuous decrease of the surface tension values due to the adsorption of surfactant molecules at the interface. The second region is located at higher surfactant concentrations where the surface tension values are almost stable. The intercept of these two regions gave the critical micelle concentration values, CMC [14] Table 1.

Commonly, the surface tension depression of the naturally obtained surfactants is relatively high. While in case of vanillin derived cationic surfactants, the surface tension reduction is comparatively low. Table 2 reveals that the surface tension values at the CMC range between 30 and 42 mNm^{-1} for octyl and hexadecyl derivatives,

Scheme 1 The synthetic route of vanillin derived cationic surfactants



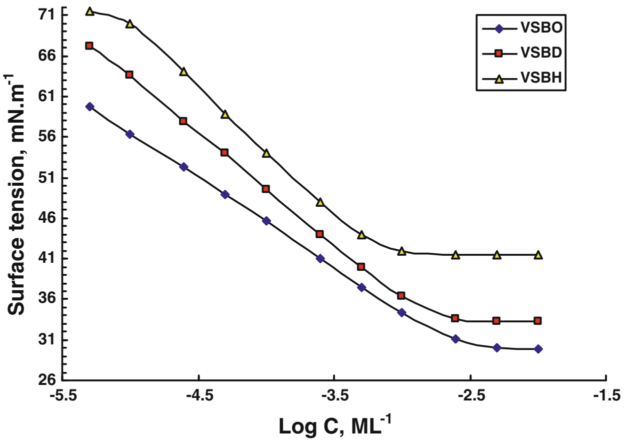


Fig. 1 Surface tension versus log C profile of the synthesized vanillin-derived cationic surfactants at 25 °C

respectively. On the other hand, the efficiency as a measurable parameter to compare the surface activity of different surfactants showed that the octyl derivative has the lowest value, i.e., low concentration of vanillin derived cationic surfactant containing the octyl chain has the ability to depress the surface tension of its solution to 52 mNm^{-1} than the derivatives containing dodecyl or hexadecyl chains.

It is clear that the critical micelle concentration values of the synthesized surfactants obtained from Fig. 1 have a descending trend by increasing the hydrophobic chain length. That shows the ability of these surfactants to micellized at relatively low concentrations. The formation of micelles is a reflection for the repulsion occurring between the different hydrophobic chains and the aqueous medium [14]. The lowest CMC value was obtained at 0.56 mM corresponding to the VSBH derivative.

The decrease in the CMC values reveal that the molecules tend to form micelles at relatively low concentrations and that is due to the mutual repulsion between the polar water molecules and the nonpolar hydrophobic chains. Higher repulsion occurred between the longer hydrophobic chain (SBVH) and the water phase, as a result, the CMC value decreased considerably [14].

The CMC values varied linearly with the variation of the hydrophobic chain length indicating that the relation between them is that in Eq. 1 [14]:

$$\mathrm{Log\ CMC} = \mathrm{A} + \mathrm{BN} \qquad (1)$$

where, A and B are constants reflecting the free energy changes involved in transferring the hydrophilic group and a methylene unit of the hydrophobic group from the aqueous phase to the micellar phase.

Figure 2 shows the effect of the hydrophobic chain lengths of the synthesized cationic surfactants at 25 °C on their critical micelle concentration values. The gradual increase in the hydrophobic chain length of the synthesized cationic surfactants gradually decreases their critical micelle concentration as listed in Table 2. The hexadecyl derivative *(VSBH)* had the lowest CMC values (0.56 mmol L^{-1}) at 25 °C compared to the other homologues.

The accumulation of surfactant molecules at the air–water interface is calculated according to the dγ/dlogC term (surface pressure) which describes the change in the surface tension by finite change of the surfactant concentration. Increasing the surface pressure of the system indicates

Table 1 Properties of the synthesized compounds

Compound	Mol. Formula	M. Wt. (gm/mole)	Yield (%)	C%		H%		N%		Br%	
				Calc.	Calc.	Calc.	Found	Calc.	Found	Calc.	Found
VSB	$C_{13}H_{12}N_2O_2$	228.25	92	68.41	67.32	5.30	5.06	12.27	12.19	–	–
OB	$C_{10}H_{19}O_2Br$	251.16	95	47.82	47.49	7.63	7.57	–	–	31.81	31.53
DB	$C_{14}H_{27}O_2Br$	307.27	95	54.72	54.34	8.86	8.80	–	–	26.00	25.82
HB	$C_{18}H_{35}O_2Br$	363.38	95	59.50	59.08	9.71	9.64	–	–	21.99	21.84
VSBO	$C_{23}H_{31}O_4N_2Br$	479.42	90	57.60	57.2	6.52	6.47	5.84	5.79	16.67	16.51
VSBD	$C_{27}H_{39}O_4N_2Br$	535.52	91	60.51	60.10	7.40	7.21	5.23	5.11	14.90	14.72
VSBH	$C_{31}H_{47}O_4N_2Br$	591.63	91	62.91	62.42	8.00	7.89	4.77	4.68	13.51	13.40

Table 2 Surface and thermodynamic properties of the vanillin derived surfactants at 25 °C

Compound	CMC, mM	γ_{IT}, mNm^{-1}	π_{cmc}, mN m^{-1}	Pc_{20}, mM	dγ/dlogC	Γ_{max}, mol m^{-2}	A_{min}, A^2	ΔG_{mic}, kJ mol^{-1}	ΔG_{ads}, kJ mol^{-1}
VSBO	2.50	4	30.5	0.026	−11.02	0.96	173.9	−29.97	−33.05
VSBD	1.51	3	34.0	0.067	−13.61	1.18	140.8	−32.91	−36.19
VSBH	0.56	2	42.0	0.135	−15.52	1.34	123.6	−36.89	−40.04

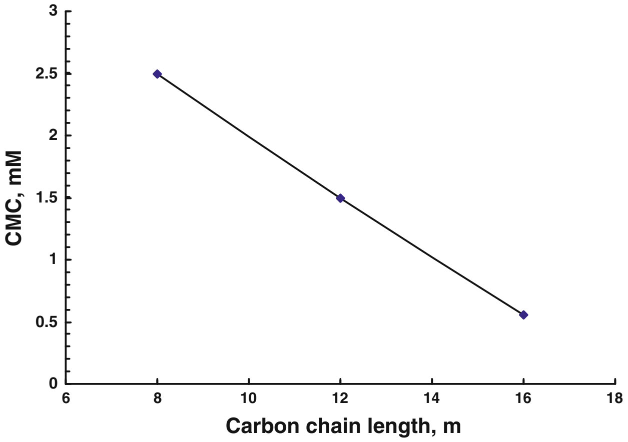


Fig. 2 Relation between CMC values and the carbon chain length of the vanillin-derived cationic surfactants

the high accumulation of surfactant molecules at the interface, Table 2. The maximum surface excess values (Γ_{max}) of the surfactant solutions were calculated (Table 2) according to Gibb's adsorption equation (Eq. 2) [15].

$$G_{max} = -(1/nRT)(d\gamma/d\ln C) \tag{2}$$

Where, $d\gamma/\mathrm{dln}\ C$ is the maximum slope and R, T, C are the gas constant, temperature and concentration.

It is clear that the maximum surface excess values are increased gradually by increasing the hydrophobic chain length. This indicates the high accumulation of the hexadecyl derivative at the interface, which indicates the maximum compactness of these molecules. As a result, the adsorbed monolayer formed is expected to be very dense [16]. The compacted and highly dense adsorbed monolayer plays an important role in several interfacial applications of surfactant solutions including emulsification, solubilization, corrosion inhibition and biological activity.

The compacted adsorbed layer restricts the motion of the surfactant molecules at the interface and forces them to be located in the minimum area, i.e. the molecules are located perpendicular to the interface. This suggests the decrease of the minimum surface area is caused by increasing the hydrophobic chain length, Table 2.

Interfacial Tension

The adsorption of surfactant molecules at the boundaries between two phases is to reduce the free energy at these boundaries. This lowering is expressed or reflected on the values of the interfacial energy. The term interfacial tension (γ_{IT}) is used instead of interfacial free energy per unit area. The tendency of molecules towards accumulation at the interfaces is a fundamental property of surfactant molecules.

In principle, the stronger the adsorption tendency is the better the surfactant. The degree of surfactant concentration at a boundary depends on several factors including: chemical structure of surfactants, nature of phases, concentration of the surfactant, chemical structure of the nonpolar phase and temperature. The interfacial tension values of the vanillin derived cationic surfactants were measured between aqueous surfactant solution (0.1% wt) and light paraffin oil at 25 °C, Table 2. Inspecting the interfacial tension data leads to two points. First, the octyl chain derivative (*VSBO*) has a high interfacial tension compared to the long chain derivatives. Second, the dodecyl and hexadecyl derivatives (*VSBD*, *VSBH*) showed low interfacial tension values at 3 and 2 mNm^{-1}, which is lower than the conventional cationic ammonium surfactant. This materializes in their applicability to interfacial applications including: corrosion inhibition, antimicrobial agents and phase transfer processes.

Thermodynamics of Adsorption and Micellization

The thermodynamic characteristics of vanillin cationic surfactants were studied using the calculated values of adsorption and micellization free energies ΔG_{ads}, ΔG_{mic}. These data were calculated using the thermodynamic Eqs. 3, 4, [10, 14] Table 2.

$$\Delta G_{mic} = 2.303\ RT\ \log(\mathrm{CMC}) \tag{3}$$

$$\Delta G_{ads} = DG_{mic} - (0.006\ X\pi_{cmc}\ X\ A_{min}) \tag{4}$$

The micellization free energies of the targeted surfactants in their aqueous solutions showed negative sign, thus indicating a spontaneous micellization process. The driving force of micelle formation is the repulsion occurring between the hydrophobic chains and the polar medium. The stabilization of the micelles formed occurred through the attraction forces between the positively charged head groups and the water molecules. The association of the counter ions (Br^-) in the attracted system also increases the stability of the micelles formed. Obviously, increasing the hydrophobic chain length increases the negativity of the ΔG_{mic} values. This enforces the idea of increasing the micellization tendency by increasing the number of methyl groups along the hydrophobic chains. The gradual increase in the negativity of ΔG_{mic} agrees with the trend of the critical micelle concentration values listed in Table 2. On the other hand, the adsorption free energy change of the studied surfactants showed an exact response as ΔG_{mic} by increasing the hydrophobic chain lengths.

The negativity of both ΔG_{ads} and ΔG_{mic} indicates that the two processes occurred spontaneously. The two values of ΔG_{mic} and ΔG_{ads} are comparable and refer to the equilibrium between the two phases of the surfactant molecules (adsorbed and micellized phase). Also, the slight increase in ΔG_{ads} values (more negative) may be ascribed to the tendency of the molecules to adsorb at the air–water interface until complete surface coverage. Beyond this, the molecules diffuse to the bulk of their solution to form micelles. Hence, the micellization and adsorption processes are governed by the thermodynamic aspects. The chemical structure of these molecules is the main factor influencing their thermodynamic aspects.

Emulsification

The tendency to enhance oil-in-water emulsion formation is one of the main characteristic applications of surface active agents. Within the different types of surfactants, anionic surfactant are generally the most useful emulsifying agents. In spite of this, some cationic surfactants play this role, especially for the formation of short-term emulsions. The emulsifying power of the synthesized surfactants was expressed as the time required for the separation of 90% of the water in the oil–water emulsion. Figure 3 reveals that there is a gradual increase in the emulsification power of the synthesized surfactants with an increase in the hydrophobic chain length. The maximum emulsification power at 1,920 s. is obtained in the presence of hexadecyl vanillin derived cationic surfactant (VSBH). The results were strongly related to the surface concentration (Γ_{max}) of these surfactants at the interface. The high surface concentration indicates a high surfactant population at the boundaries between the oil and water; this consequently provides more stability to the emulsions formed.

Biodegradability Assessment

Biodegradation is the most important characteristic for the irreversible removal of chemicals from aquatic and global environments. It may be defined as the destruction of chemical compounds by the biological action of living organisms. The biodegradability of the derived surfactants was evaluated by applying the Modified Screening Test [12]. In this test, the final biodegradation or mineralization of the surfactants (i.e., the microbial transformation of the parent chemical into final products of the degradation process, such as carbon dioxide and water) was evaluated. In the course of the biodegradation test, the DOC (dissolved organic carbon) concentrations were determined at the beginning and at regular time intervals for a 28 day period. Biodegradation was stated as the percentage of DOC removal within 28 days (Fig. 4).

Biodegradation percentages of approximately 81% after day 28 are exceeded; the specified biodegradation passes the level in this test (70%), allowing us to classify the synthesized cationic surfactants as readily biodegradable compounds, i.e., eco-friendly compounds.

These good results contrast with the low biodegradation level of the classic cationic surfactants of quaternary ammonium salts [17]. In fact, the chemical structures of the synthesized cationic surfactants have been designed from a naturally occurring compound (vanillin) which has the ability to degrade by the action of the environmental microorganisms. The simplest pathway of the degradation considered in case of our compounds is that the microorganisms attach themselves to the vanillin moiety in the chemical structure and then the fatty alkyl chains follow the pathway of chain-shortening through the *β*-oxidation pathway [18] and finally, the microorganisms completely degrade the hydrocarbon chain. The alkyl chain length does not affect the biodegradation level; even a surfactant with 16 carbon atoms in the molecule (which gives it a high hydrophobic character) exceeds the biodegradation level in 21 days.

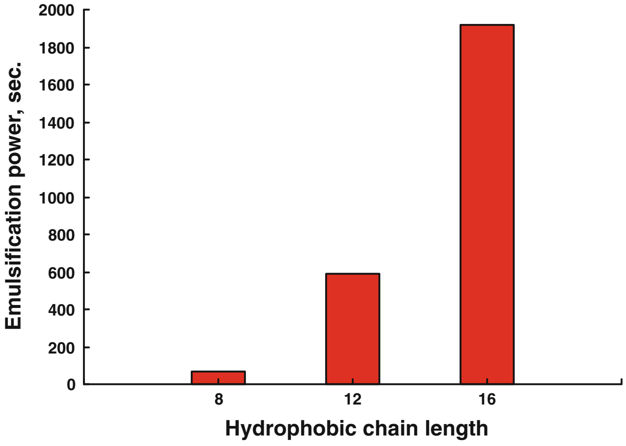


Fig. 3 Effect of hydrophobic chain length on the emulsification power of the vanillin-derived cationic surfactants

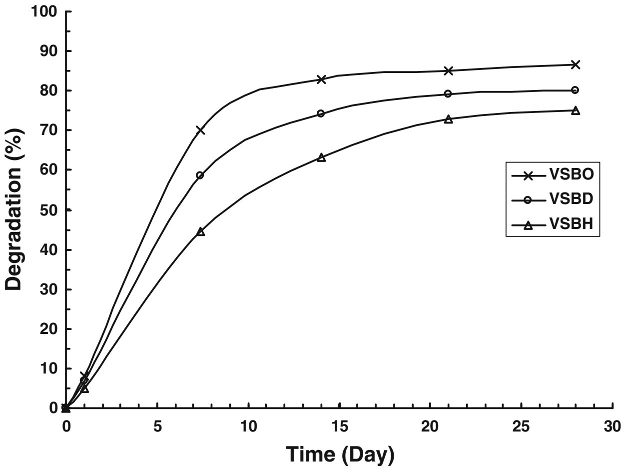


Fig. 4 Biodegradation of the synthesized cationic surfactants

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Nabel A. Negm His Ph.D. dissertation in 2000 at Ain Shams University in Cairo, Egypt, was on the synthesis and evaluation of some quaternary ammonium surfactants. He is now a professor of applied organic chemistry at the Petrochemicals Department of the Egyptian Petroleum Research Institute. He is interested in several fields of surfactant applications, including detergency, emulsification, solubilization, corrosion inhibition of metals, biological activity of surfactants and their metal complexes towards bacteria, fungi and yeasts, and the interaction of different types of surfactants with proteins and macromolecules.

Nadia G. Kandile received her Ph.D. in 1975 from Ain Shams University in Cairo, Egypt, where she is currently a professor of applied and environmental chemistry, after being a professor of organic chemistry. Her research and development activities deal with designing new chemical compounds using simple technology, environmental problems in the petroleum sector, industrial waste water treatment and drug agents such as those which are antimicrobial-antifungal and anticancer. She is a visiting Professor for the Surfactant Institute in Norman OK (USA) on a project on the prevention of oil pollution by fluorinated surfactants. She is a Titular Member of the IUPAC Chemistry & Environment division.

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