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Investigating the potential of using acoustic frequency on the degradation of linear alkylbenzen sulfonates from aqueous solution*

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Abstract: The effectiveness of using acoustical (sonochemical) reactor for degradation of linear alkylbenzen sulfonate (LAS) from aqueous solution was investigated. LASs are anionic surfactants, found in relatively high amounts in domestic and industrial wastewaters. In this study, experiments on LAS solution were performed using methylene blue active substances (MBAS) method. The effectiveness of acoustical processor reactor for LAS degradation is evaluated with emphasis on the effect of treatment time and initial LAS concentration. Experiments were performed at initial concentrations of 0.2, 0.5, 0.8 and 1.0 mg/L, acoustic frequency of 130 kHz, applied power of 500 W and temperature of 18 °C~20 °C. At the conditions involved, LAS degradation was found to increase with increasing sonochemical time. In addition, as the concentration increased, the LAS degradation rate decreased in the acoustical processor reactor.

Key words: Acoustical reactor, Linear alkylbenzen sulfonate (LAS), Acoustic frequency, Treatment time, Power
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INTRODUCTION

Industries worldwide discharge a wide range of surfactant, or surface-active agents, to their wastewater treatment facilities. Water pollution caused by synthetic surfactants has been increasing during the past few years due to their extensive use in household, agriculture and other cleaning operations. Synthetic surfactants released into the aquatic system have adversely affected ecosystems (Baleux and Caumette, 1977). Today the detergent wastes constitute a major component of organic pollutants that are carried by various means into lakes, rivers, and seas and cause serious environmental problem (Eganhouse and Kaplan, 1982; Baskaran *et al*., 1991; Zeni and Caligiuri, 1992; Takada *et al*., 1992; Abd-Allah, 1995).

A surfactant combines in a single molecule—a strongly hydrophobic group with a strongly hydrophilic one. Such molecules tend to congregate at the interfaces between the aqueous medium and the other phases of the system such as air, oily liquids, and particles, thus imparting properties such as foaming, emulsification, and particle suspension (APHA, 2005).

Synthetic detergents are a mixture of linear alkylbenzene sulfonate (LAS) and its isomers together with other additives. LAS is a surface-active material found in relatively high amounts in domestic and industrial wastewaters, discharged mainly from the textile, cosmetic and tanning industries (Comninellis, 1994; Lin and Peng, 1994; Brillas *et al*., 1995).

LASs are anionic surfactants (Fig.1), which were introduced in 1964 as the readily biodegradable replacement for highly branched tetrapropylbenzenesulfonate (ARCRCP, 2005). LAS contain an aromatic

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ring sulfonated at the para position and attached to a linear alkyl chain at any position except the terminal ones. The commercial product mainly consists of a complex mixture of various homologues and isomers, representing different alkyl chain lengths (ranging from 10 to 14 carbon atoms) and aromatic ring positions along the linear alkyl chain (Di Corcia *et al.*, 1999; Scott and Jones, 2000).

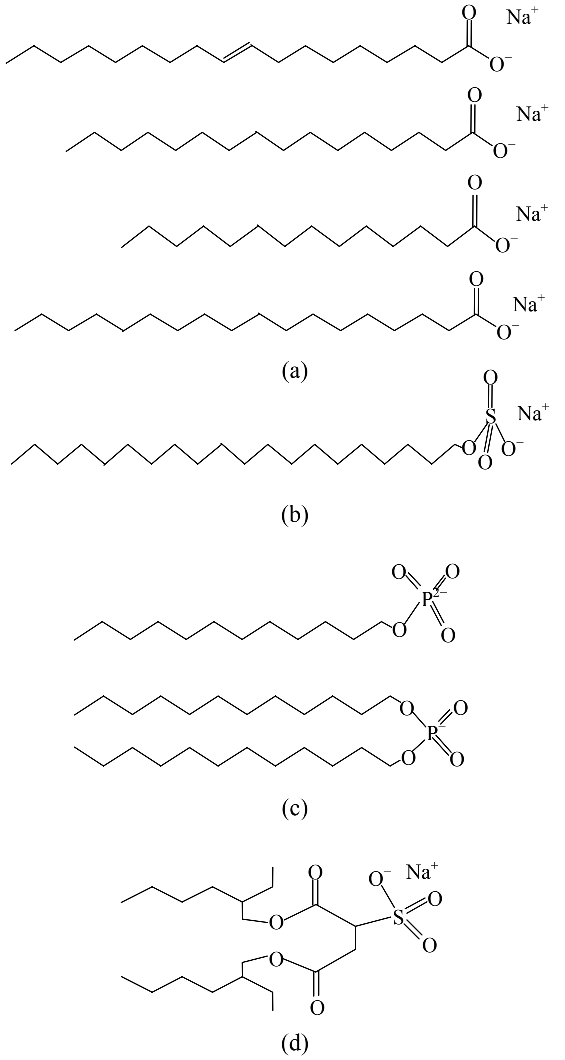

Fig.1 Anionic surfactants. (a) Some common soaps (from the top down): sodium oleate, sodium palmitate, sodium myristate and sodium stearate; (b) Sodium dodecyle sulfate, one of the most common of the alkyl sulfate style of anionic surfactants; (c) Two alkyl phosphates: an alkyl sulfonate and an alkyl benzene sulfonate; (d) Sodium di(2-ethylhexyl) sulfosuccinate

Their presence in sewage works is variable depending on their use in industrial processing in addition to domestic activities. An average LAS concentration of 1~10 mg/L can be found in municipal wastewater treatment dealing only domestic wastewater (Field *et al.*, 1995) but this range is noticeably increased when industrial wastes from washing processes are also treated (Beltrán *et al.*, 2000).

Sonochemical/acoustical mechanisms

In chemistry, the study of sonochemistry is concerned with understanding the effect of sonic waves and wave properties on chemical systems. Sonochemistry is the application of ultrasonics to chemical reactions and processes (Mason, 1999; Crum *et al.*, 1999). Ultrasonics is the science of sound waves above the limits of human audibility. The frequency of a sound wave determines its tone or pitch. Low frequencies produce low or bass tones. High frequencies produce high or treble tones (Suslick and Price, 1999). Ultrasonics is a sound with a pitch so high that the human ear cannot hear it. Frequencies above 18 kHz usually considered to be ultrasonic. The frequencies used for ultrasonic cleaning range from 20000 kHz to over 100000 kHz (Gong and Hart, 1998). Rarefaction and compression cycles induced by the ultrasonic pulses in the liquid generate cavities that grow until they reach a critical size and then due to resonant absorption of energy the bubbles undergo a violent collapse. The fast implosion of the cavitation bubbles produces a quasi-adiabatic heating of the vapor phase inside the cavity that yields localized high temperatures (thousands of K) and pressures (Leighton, 1994; Neppiras, 1980). Water molecules under such extreme conditions undergo thermal dissociation to yield H· and OH· radicals. Organic solutes in the vicinity of a collapsing bubble or partitioned into the gas phase of the bubble undergo thermal decomposition and/or react with the reactive radicals (Mason, 2003; Mason and Tiehm, 2001).

Acoustic cavitation (frequency) process

Irradiation by acoustical or sonochemical reactor in a liquid leads to the acoustic cavitation process, such as the formation, growth, and collapse of bubbles, accompanied by the generation of local high temperature, pressure, and reactive radical species. Thus, the acoustic cavitation in an aqueous solution results in chemical effects by the acoustical reactor. Three different reaction sites in the cavitation bubble, i.e., the inside of the cavitation bubbles, the gas-liquid interfacial region of the cavitation bubbles, and the bulk solutions are present during the sonochemical process (Mason *et al*., 1993; Yim *et al*., 2002; Adachi *et al*., 1990).

Surface-active molecules accumulate at interfaces, particularly at the water-air interface of the cavitation bubbles, and introduce major physical and chemical changes to their sonochemical response. Since cavitation bubbles are the "hot spots" where sonochemical reactions take place, surfactants should be particularly a good target for this treatment (Weavers *et al*., 2005; Manousaki *et al*., 2004; Destaillats *et al*., 2000).

MATERIALS AND METHODS

General principle

An anionic detergent, LAS, was chosen as model detergents. LAS can be analyzed by nonspecific methods. The assay usually used is one for substances that react with methylene blue, which responds to any compound containing an anionic and hydrophobic group. It thus suffers from analytical interference if used for environmental samples; furthermore, the sensitivity of this method is about 0.02 mg/L. Although nonspecific alternatives to this method have been developed, they are not commonly used. Specific methods for environmental analysis are available only for LAS. An improved method based on methylene blue reactivity and high-performance liquid chromatography is available for analysis of LAS.

Methylene blue active substances (MBAS) bring about the transfer of methylene blue, a cationic dye, from an aqueous solution into an immiscible organic liquid upon equilibration. This occurs through ion pair formation by the MBAS anion and the methylene blue cation. The intensity of the resulting blue color in the organic phase is a measure of MBAS. Anionic surfactants are among the most prominent of many substances, natural and synthetic, showing methylene blue activity.

The MBAS method is useful for estimating the anionic surfactant content of waters and wastewaters, but the possible presence of other types of MBAS always must be kept in mind. This method is relatively simple and precise. It comprises three successive extractions from acid aqueous medium containing excess methylene blue into chloroform, followed by an aqueous backwash and measurement of the blue color in the chloroform by spectrophotometry at 453 nm. The method is applicable at MBAS concentrations down to about 0.025 mg/L.

Apparatus and procedures

Spectrophotometer: for use at 453 nm, providing a light path of 1 cm.

Separatory funnels: 500 ml, preferably with inert TFE stopcocks and stoppers.

Acoustical reactor: An acoustical reactor (Basin-Batch) operating at a fixed frequency of 130 kHz and a power of 500 W was used for sonochemical experiments (Fig.2). Both frequency and power were adjusted in a generator connected to the transducer. Reactions were carried out in a stainless steel vessel, which was closed during sonication. The vessel was immersed in a water bath. In all experiments, 200 ml of detergent aqueous solution was prepared daily and exposed in acoustical reactor. On the other hand, 200 ml solution was sonicated with the sound waves emitting from the bottom of the acoustical reactor.

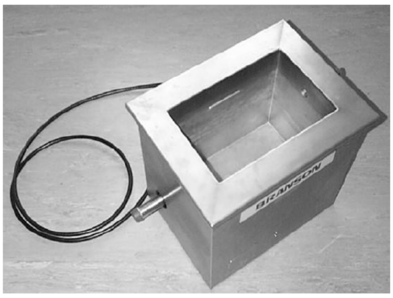

Fig.2 Acoustical processor reactor for LAS degradation

Chemicals

Stock LAS solution: Weigh an amount of the reference material equal to 1.00 g LAS on a 100% active basis. Dissolve in water and dilute to 1000 ml. Store in a refrigerator to minimize biodegradation.

Standard LAS solution: Dilute 10.00 ml stock LAS solution to 1000 ml with water.

Phenolphthalein indicator solution, alcoholic.

Sodium hydroxide, NaOH, 1 mol/L.

Sulfuric acid, 1 and 6 mol/L.

Chloroform.

Methylene blue reagent: Dissolve 100 mg methylene blue in 100 ml water. Transfer 30 ml to 1000 ml flask. Add 500 ml water, 41 ml 6 mol/L sulfuric acid, and 50 g sodium phosphate, monobasic, monohydrate.

Wash solution: Add 41 ml 6 mol/L sulfuric acid to 500 ml water in a 1000 ml flask. Add 50 g $NaH_2PO_4 \cdot H_2O$ and shake until dissolved. Dilute to 1000 ml.

Methanol.

Hydrogen peroxide.

Glass wool: Pre-extract with $CHCl_3$ to remove interferences.

Water, reagent-grade, MBAS-free. Use for making all reagents and dilutions.

Preparation of calibration curve: Prepare a series of separatory funnels with 0, 1.00, 3.00, 5.00, 7.00, 9.00, 11.00, 13.00, 15.00 and 20.00 ml standard LAS solution. Add sufficient water to make the total volume 100 ml in each separatory funnel. Plot a calibration curve of absorbance vs micrograms LAS taken, specifying the molecular weight of the LAS used.

Sample size: For direct analysis, select sample volume on the basis of expected MBAS concentration as shown in Table 1.

Table 1 Expected MBAS concentration

Sample taken (ml)	Expected MBAS concentration (mg/L)
400	0.025~0.080
250	0.08~0.40
100	0.4~2.0

Peroxide treatment: To avoid decolorization of methylene blue by sulfides, add a few drops of 30% hydrogen peroxide.

Extraction:

(1) Add sample to a separatory funnel. Make alkaline by dropwise addition of 1 mol/L NaOH, using phenolphthalein indicator. Remove pink color by dropwise addition of 1 mol/L sulfuric acid.

(2) Add 10 ml $CHCl_3$ and 25 ml methylene blue reagent. Rock funnel vigorously for 30 s. and let phases separate. Alternatively, place a magnetic stirring bar in the separatory funnel; lay funnel on its side on a magnetic mixer and adjust speed of stirring to produce a rocking motion. Excessive agitation may cause emulsion formation. To break persistent emulsions add a small volume of isopropyl alcohol; add same volume of isopropyl alcohol to all standards. Some samples require a longer period of phase separation than others. Before draining $CHCl_3$ layer, swirl gently, and then let settle.

(3) Draw off $CHCl_3$ layer into a second separatory funnel. Rinse delivery tube of first separatory funnel with a small amount of $CHCl_3$. Repeat extraction two additional times, using 10 ml $CHCl_3$ each time. If blue color in water phase becomes faint or disappears, discard and repeat, using a smaller sample.

(4) Combine all $CHCl_3$ extracts in the second separatory funnel. Add 50 ml wash solution and shake vigorously for 30 s. Emulsions do not form at this stage. Let settle, swirl, and draw off $CHCl_3$ layer through a funnel containing a plug of glass wool into a 100 ml volumetric flask; filtrate must be clear. Extract wash solution twice with 10 ml $CHCl_3$ each time and add to flask through the glass wool. Rinse glass wool and funnel with $CHCl_3$. Collect washings in volumetric flask, dilute to mark with $CHCl_3$, and mix well.

(5) Measurement: Determine absorbance at 453 nm against a blank of $CHCl_3$.

Calculation: From the calibration curve read micrograms of apparent LAS corresponding to the measured absorbance:

$$\text{mg MBAS/L} = \mu\text{g apparent LAS/ml original sample.}$$

All the analyses were performed according to the procedures outlined in standard methods (APHA, 2005).

RESULTS AND DISCUSSION

In this research, sonodegradation of LAS was applied for different periods of time: 20, 30, 40, 50, 60, 70, 80, 90, 100, 110 and 120 min. Also, sonodegradation experiments of LAS were carried out in the presence of various concentrations to observe if there was any effect on the degradation of LAS. Sonodegradation of LAS at an initial concentration of 0.2, 0.5, 0.8 and 1 mg/L, acoustic frequency 130 kHz, temperature 18 °C~20 °C and power 500 W was investigated. The experiments were performed at pH 6.8~7.0.

LAS concentration of 0.2 mg/L provided better LAS degradation than 1 mg/L for all sonication times. Increasing the concentration from 0.2 mg/L to 1.0

mg/L showed a decrease in degradation of LAS. About 92.35% degradation of LAS was observed with concentration of 0.2 mg/L and 71.60% degradation of LAS occurred with concentration of 1 mg/L after 120 min of sonochemical treatment. Also, experiments were conducted at various times to see if there was any synergistic effect on the degradation of LAS. Experiments showed that with acoustic reactor, about 92.35%, 89.21%, 88.11% and 71.60% degradation of LAS occurred after 120 min but only 74.36%, 39.00%, 32.28% and 28.29% degradation of LAS was observed after 20 min as shown in Fig.3.

Statistical analysis

The potential of using acoustic frequency on the degradation of LAS was analyzed statistically by using *t*-test, Spearman's rho test, one-way test and post-hoc test. The variables were treatment time, *T* (Table 2), degradation percentage (*DP*), and initial concentration, *C* (Table 3). Frequency and power were fixing parameters. In this statistical analysis, Spearman's rho test (nonparametric correlations) shows that correlation is significant at the 0.01 levels (Table 4). Also, post-hoc tests (multiple comparisons) show that mean difference is significant at the 0.05 levels (Table 5).

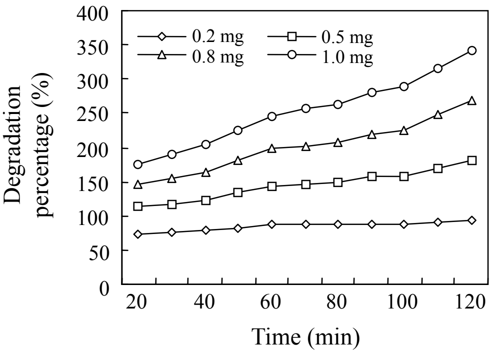

Fig.3 Degradation percentage of LAS vs treatment time for different concentrations

Effect of concentration

The sonodegradation of various concentrations of LAS surfactant at 130 kHz was investigated. The experiments were carried out at four intervals. The effect of concentration was studied in researches. At low concentration, surfactants preferentially accumulate on cavitation bubble surfaces, reducing the surface tension of the solution. In addition, at high concentrations when micelles are present, the rate constant increases slightly over that at slightly lower concentration (Weavers *et al.*, 2005). The sono-

Table 2 Frequency and percent (treatment time, *T*)

Valid	Frequency	Percent (%)	Cumulative percent (%)
20.00	4	9.1	9.1
30.00	4	9.1	18.2
40.00	4	9.1	27.3
50.00	4	9.1	36.4
60.00	4	9.1	45.5
70.00	4	9.1	54.5
80.00	4	9.1	63.6
90.00	4	9.1	72.7
100.00	4	9.1	81.8
110.00	4	9.1	90.9
120.00	4	9.1	100.0
Total	44	100.0	

Table 3 Frequency and percent (initial concentration, *C*)

Valid	Frequency	Percent (%)	Cumulative percent (%)
0.20	11	25.0	25.0
0.50	11	25.0	50.0
0.80	11	25.0	75.0
1.00	11	25.0	100.0
Total	44	100.0	

Table 4 Spearman's rho-test (nonparametric correlations)

C			*T*		
Correlation coefficient	Sig. (2-tailed)	*N*	Correlation coefficient	Sig. (2-tailed)	*N*
-0.602^*	0.000	44	0.698^*	0.000	44

*Correlation is significant at the 0.01 levels (2-tailed)

Table 5 One-way test (initial concentration, *C*)

	Sum of squares	*df*	Mean square	*F*	Sig.
Between groups	7091.813	3	2363.938	12.082	0.000
Within groups	7826.633	40	195.666	–	–
Total	14918.446	43			

chemical degradation of LAS at various concentrations indicated that removal rates decrease with increasing LAS concentration ($P<0.001$) as shown in Table 6. On the other hand, a significant reduction in LAS degradation occurred after 120 min ($SD=18.62634$) as shown in Table 7.

Effect of treatment time

In order to observe the effect of treatment time on the LAS degradation rate during treatment, sonodegradation or treatment time test for aqueous LAS concentration was done at eleven intervals. As clearly seen, by increasing the treatment time, considerable levels of LAS degradation can be expected after 120 min, as shown in Table 8 ($P=0.012$). On the other hand, it was observed that the degradation efficiency of acoustic frequency was increased when treatment time was increased. This effect is due to the increase in the exposure time between the LAS solution and the acoustic cavitation process as the time of sonication was increased (Jyoti and Pandit, 2003).

Table 6 Post-hoc test (multiple comparisons of concentrations)

I_C	J_C	I_C–J_C	Sig.	95% confidence interval	
				Lower bound	Upper bound
0.20	0.50	24.9264*	0.000	12.8716	36.9811
	0.80	28.2745*	0.000	16.2198	40.3293
	1.00	32.6727*	0.000	20.6180	44.7275
0.50	0.20	−24.9264*	0.000	−36.9811	−12.8716
	0.80	3.3482	0.578	−8.7066	15.4029
	1.00	7.7464	0.201	−4.3084	19.8011
0.80	0.20	−28.2745*	0.000	−40.3293	−16.2198
	0.50	−3.3482	0.578	−15.4029	8.7066
	1.00	4.3982	0.465	−7.6566	16.4529
1.00	0.20	−32.6727*	0.000	−44.7275	−20.6180
	0.50	−7.7464	0.201	−19.8011	4.3084
	0.80	−4.3982	0.465	−16.4529	7.6566

I_C: initial concentration level; J_C: secondary concentration level; I_C–J_C: mean difference of degradation (%); *The mean difference is significant at the 0.05 levels

Effect of power

The positive effect of power on the sonodegradation of LAS may be explained in terms of cavitational activity. High levels of acoustic power increase the number of cavitational events and consequently the opportunities for free radicals to be generated enhance degradation (Psillakis *et al*., 2004; Thompson and Doraiswamy, 1999).

CONCLUSION

Acoustical reactor at a frequency 130 kHz is capable to some degree of LAS degradation in aqueous synthetic solutions. Potential of acoustical reactor for LAS degradation was evaluated with emphasis on the effect of treatment time and initial LAS concentration. Experiments showed that treatment time is one of the most important parameters for LAS degradation. Also, this study indicates that the overall treatment efficiency rises with decreasing LAS concentration.

Acoustical reactors alone may not be useful for reducing completely complex wastewaters of high surfactant load. In this respect, effectiveness may be improved by coupling acoustical reactors with other treatment processes including ozone, UV, chlorination and H_2O_2. Alternatively, acoustical processor reactor could be used as a pre-treatment stage in a sequential chemical and biological treatment process.

Table 7 *t*-test (treatment time, *T*)

Parameter	Value
Power	500
N	44
Mean	63.3561
SD	18.62634
Min.	28.29
Max.	92.35

Table 8 One-way test (treatment time, *T*)

	Sum of squares	*df*	Mean square	*F*	Sig.
Between groups	6884.803	10	688.480	2.828	0.012
Within groups	8033.643	33	243.444	–	–
Total	14918.446	43			

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