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SONOLUMINESCENT SPECTROSCOPY WITH SONOLUMINESCENCE INITIATION BY ULTRAHIGH-FREQUENCY ULTRASOUND: DETECTING THE CONTENT OF THE MAIN SUBSTANCE IN HIGHLY SATURATED SOLUTIONS

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This paper presents further development of sonoluminescence spectroscopy, a novel method of chemical analysis. In particular, the application of ultrahigh-frequency (≥10.0 MHz) ultrasound for the initiation of sonoluminescence was considered for detecting the content of the main substance in natural brines and highly concentrated technological solutions. The influence of the initiating ultrasound frequency on the intensity of sonoluminescence of CsCl, LiCl, and NaCl was established. The identity of the processes of cavitation bubble formation and collapse in solutions of alkali metal chlorides was demonstrated. It was shown that the maximum intensity of sonoluminescence for alkali metal chlorides is reached at an ultrasound frequency of 20–22 MHz. A technique has been developed for determining the content of Cs, Li, and Na in highly concentrated technological solutions and brines using ultrahigh-frequency ultrasound to initiate sonoluminescence. The validity of the method was verified by analyzing the same samples with alternative methods: gravimetry and sonoluminescence spectroscopy using both high- and low-frequency ultrasound for sonoluminescence initiation.

Keywords: sonoluminescence, cavitation bubbles, highly concentrated solutions, natural brines, ultrasound, relative standard deviation.

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Introduction

The basis of the sonoluminescence spectroscopy method of chemical analysis is the physical phenomenon of sonoluminescence – the emission of light that occurs in a liquid under the action of cavitation processes. This phenomenon arises as a result of heating of the vapor–gas mixture inside a cavitation bubble during its rapid shrinkage or collapse: the temperature at certain points inside the bubble can reach 10,000^{0}C, while the average temperature is about 2,700^{0}C [1–5]. The sonoluminescence spectrum consists of the following components [6–10]:

1) a band corresponding to the emission of an excited water molecule, $H_2O^* \rightarrow H_2O + h\nu$ (265–280 nm);

2) a band originating from the emission of the excited hydroxyl radical, $OH^* \rightarrow OH^- + h\nu$ (300–315 nm), as well as lines of alkali and alkaline earth metals;

3) chemiluminescence of some organic compounds formed along with radical products of water splitting;

4) bands associated with the emission of metals.

Analytical chemistry currently employs components 1, 3, and 4 of the sonoluminescence spectrum [1,4,6]. Among these, the method of sonoluminescence spectroscopy based on the emission lines of metals has become the most widely used. Researchers in [1,6–13] studied sonoluminescence as a source of an analytical signal: the emission spectra of individual elements.

It was experimentally determined that individual sonoluminescence spectra can be obtained only for elements whose metallic state is characterized by a boiling point of about 2,700^{0}C and an ionization energy of approximately 7.65 eV [1,6].

Sonoluminescence spectroscopy is one of the most effective methods for determining the content of the main component in highly concentrated technological solutions and natural brines [11–13]. When low-frequency (LF) ultrasound (20–100 kHz) is applied to induce sonoluminescence, the determination of the main component in highly concentrated technological solutions (400–600 g/dm^3) yields results with a relative standard deviation (S_r) in the range of 0.092–0.141, depending on the concentration of the substance being determined [11].

When high-frequency (HF) ultrasound (500 kHz–5 MHz) is used to induce sonoluminescence, the determination yields results with a relative standard deviation of 0.056–0.080, depending on the concentration of the determined substance [14].

The application of sonoluminescence spectroscopy for determining the content of the main component in underground sodium chloride brines (at depths of 300–350 m) has been reported [1]. High-frequency ultrasound with a frequency of 500 kHz–5.0 MHz was used to induce sonoluminescence. In comparison with low-frequency ultrasound (18–100 kHz), the metrological characteristics of the analysis improved significantly. Specifically, the relative standard deviation, S_r, for determining the main component in underground brines decreased from 0.091–0.140 to 0.055–0.082.

The improvement in the metrological characteristics of the analysis results for determining the main component in highly concentrated technological solutions (400–600 g/dm^3) and natural brines when using HF ultrasound to initiate sonoluminescence, compared with LF ultrasound, can be explained by:

1) the higher stability of piezoelectric ultrasonic emitters relative to magnetostrictive ones;

2) differences in the formation and collapse of cavitation bubbles under HF ultrasound, where larger bubbles with a smaller total volume are produced compared with LF ultrasound.

The latter factor leads to a substantial (20–100-fold) reduction in the sensitivity of determining the main component in highly concentrated solutions and, consequently, to a lower relative standard deviation in the analysis of solutions with concentrations ≥400 g/dm^3 [11,13].

Rapid and accurate determination of the main component content in highly concentrated solutions is of great importance for the nuclear power sector and other industries. In nuclear power plants, it is directly related to the safety and efficiency of nuclear reactors that employ cooling systems based on salt coolants such as «CsCl-1» (600 g/dm^3), «CsCl-2» (400 g/dm^3), and «LiCl» (400 g/dm^3) [1,2,14]. In the salt industry, it is essential to maintain the effective thermophysical performance of vacuum evaporation units.

This work is devoted to the further development of sonoluminescence spectroscopy and explores the possibility of using sonoluminescence spectroscopy with the initiation of the sonoluminescence by ultrahigh-frequency (UHF) ultrasound to determine the content of the main component in highly concentrated technological solutions and natural brines.

Experimental

A sonoluminescence spectrometer based on an AAS-3 atomic absorption spectrometer (Germany) was employed [1,11]. In this setup, a reactor with piezoelectric emitters for ultrasound generation was used instead of a burner or graphite furnace. Ultrasonic oscillations were generated by piezoelectric emitters operating at frequencies of 10, 15, 18, 22, and 25 MHz, connected to a 24-UZGI-K-1.6 M tube generator, which allows adjustment of ultrasound frequencies from 10 to 25 MHz [6]. Certified piezoelectric emitters manufactured by the experimental machine-building plant of the Ukrainian Research Institute of the Salt Industry were used.

The solutions were saturated with high-purity argon. Chemically pure sodium chloride for spectral analysis was used. Other reagents were of analytical grade. The solutions were prepared using distilled water.

Methodology of the experiment

Synthetic sodium chloride brines with NaCl concentrations of 200, 400, and 600 g/dm^3 (analogues of underground brines from the Drohobych deposit) were prepared in the reactor under a pressure of 2.5 atm [1,11]. Salt coolants used in nuclear power plants, «CsCl-1», «CsCl-2», and «LiCl», with main component concentrations of 400, 600, and 400 g/dm^3, respectively, were maintained in the reactor under the same pressure [14]. Natural brines from the Slavyansk and Drohobych deposits (Ukraine) were also handled in the reactor under a pressure of 2.5 atm.

To study the dependence of the sonoluminescence intensity of sodium, lithium, and cesium chlorides on the frequency of the initiating ultrasound, solutions of CsCl, LiCl, and NaCl with concentrations of 100 and 200 g/L, as well as a LiCl solution at 600 g/L, were additionally used.

A 1000 mL portion of the analyzed solution, under a pressure of 2.5 atm, was introduced into a

1200 mL chamber and saturated with argon for 5 min. The solution was then cooled to 20±1^{0}C. The analyzed solutions were continuously saturated with argon (flow rate 20 mL/min) for 0.5 h. The parameters were influenced by ultrasound. The sonoluminescence spectrometer was adjusted to the appropriate analytical lines of Na, Cs, and Li as described in [11,14], and the content of the main component was determined. During the experiments, the argon supply was maintained to prevent degassing of the solution. Experiments on the effect of ultrasound frequency on the sonoluminescence intensity were carried out using the maximum possible ultrasound intensity of 20 W/cm, limited by the capabilities of the equipment, including the mechanical strength of the ultrasound emitter [14].

The contents of CsCl and LiCl in the salt coolants of NPPs, «CsCl-1», «CsCl-2», and «LiCl», were determined using the calibration graph method. Calibration solutions were prepared from analytically pure reagents by dissolving them in distilled water in a pressure reactor at 2.5 atm [11,14].

The calibration graph method was also applied to the analysis of sodium chloride brines from the Slavyansk and Drohobych deposits, as well as synthetic NaCl brines. In this case, calibration solutions were prepared from analytically pure reagents for spectral analysis by dissolving them in distilled water in a reactor under a pressure of 2.5 atm. For the analysis of natural brines from the Slavyansk and Drohobych deposits, the same amounts of humic and fulvic acids present in these brines were added to the calibration solutions. Specifically, for the Slavyansk brines, humic acids were added at 0.014 mg/L and fulvic acids at 1.07 mg/L, whereas for the Drohobych brines, humic acids were added at 0.035 mg/L and fulvic acids at 8.74 mg/L [11,14].

Results and discussion

It was found in sonoluminescence spectroscopy that the frequency of ultrasound used to initiate sonoluminescence significantly affects the intensity of sonoluminescence of CsCl, LiCl, and NaCl (Table 1). The intensity of sonoluminescence for these chlorides changed as the ultrasound frequency was varied from 10 MHz to 25 MHz. When the frequency increased from 10 to 15 MHz, the sonoluminescence intensity of all studied chlorides decreased sharply. Subsequently, the intensity steadily increased for all chlorides, reaching a maximum at 20–22 MHz, and then decreased sharply again. This behavior indicates that the processes of formation and collapse of cavitation bubbles are similar in solutions of all alkali metal chlorides [1,8–10].

Table 1 also shows that the dependence of sonoluminescence intensity on analyte concentration in the range of 100–600 g/L is linear when ultrasound with frequencies of 18–25 MHz is used. In contrast, when ultrasound at 10 or 15 MHz is applied, the intensity vs. concentration dependence at ultra-high concentrations (600 g/L) is non-linear.

It should also be noted that the sonoluminescence intensity of NaCl brines from different deposits differs from that of synthetic brines. This can be explained by the following: 1) variations in NaCl concentration in brines from different deposits (387–406 g/dm^3); 2) the presence of organic impurities, humic and fulvic acids, in natural brines, which affect the sonoluminescence intensity. At the same time, the sonoluminescence intensity of the less concentrated brine from the Drohobych deposit (mineralization: 389 g/dm^3; average humic acid content: 0.035 mg/dm^3; fulvic acid content: 8.74 mg/dm^3) is higher than that of the Slavyansk deposit (mineralization: 408 g/dm^3; average humic acid content: 0.014 mg/dm^3; fulvic acid content: 1.07 mg/dm^3) [1].

The sonoluminescence intensity of CsCl, LiCl, and NaCl increased with rising ultrasound intensity, reaching its maximum at 20 W/cm^2, which is constrained by the mechanical strength of currently available piezoelectric emitters [1] (Table 2).

Thus, the potential of using UHF ultrasound in sonoluminescence spectroscopy for the determination of the main component in highly concentrated technological solutions and brines was demonstrated, and the relative standard deviation of the analysis results was found to depend on the ultrasound frequency (Table 3).

From Table 3, it can be seen that increasing the frequency of ultrasound used to initiate sonoluminescence improves the reproducibility and accuracy of determining the analyte concentration. However, a higher ultrasound frequency also reduces the sensitivity of the determination and shifts the linear range of the analyte concentration vs. sonoluminescence intensity dependence toward higher concentrations (Table 1). This results in improved reproducibility and accuracy for high-concentration analytes. Conversely, lower ultrasound frequencies increase the sensitivity of the determination, but for high analyte concentrations (>200 g/L), the measurements fall within the nonlinear region of the sonoluminescence intensity vs. concentration dependence [1], which reduces reproducibility and accuracy.

The optimal ultrasound frequency is 20–22 MHz, at which the best balance between sonoluminescence intensity and the operational stability of the piezoelectric ultrasound emitter is observed (Tables 1–3) [1,11].

Thus, a method for determining the main component in highly concentrated solutions of alkali metal chlorides and in natural NaCl brines has been developed. The accuracy of this method was verified by analyzing the same samples using the gravimetric method (Table 3). Sonoluminescence spectroscopy employing UHF ultrasound to initiate sonoluminescence provides improved metrological characteristics compared to the method using LF or HF ultrasound. While the gravimetric method exhibits nearly the same metrological performance as UHF sonoluminescence spectroscopy, it is less suitable for rapid analysis.

Conclusions

The use of ultra-high frequency ultrasound to initiate sonoluminescence in sonoluminescence spectroscopy was investigated. Ultrasound with frequencies of 10–25 MHz was applied to initiate sonoluminescence for determining the main component in natural brines and highly concentrated technological solutions, including salt coolants of nuclear power plants: «CsCl-1», «CsCl-2», and «LiCl», with main component concentrations of 400, 600, and 400 g/dm^3, respectively.

Methods for determining Cs, Li, and Na in highly concentrated technological solutions and natural brines were developed. The relative standard deviation of the results decreases with increasing ultrasound frequency up to 20–22 MHz at an intensity of

Table 1

Dependence of the sonoluminescence intensity of sodium, lithium, and cesium chlorides on the frequency of the initiating ultrasound*

Sample and solution concentration, g/dm^3	Sonoluminescence intensity (arbitrary units) at different ultrasound frequency					
	10 MHz	15 MHz	18 MHz	20 MHz	22 MHz	25 MHz
CsCl solution, 100	19.54	1.50	4.05	7.92	8.80	1.22
CsCl solution, 200	40.00	3.11	8.10	15.85	17.61	2.50
Coolant "CsCl-1", 400	80.02	6.23	16.21	31.71	35.23	5.01
Coolant "CsCl-2", 600	118.11	9.22	24.31	47.52	52.84	7.52
LiCl solution, 100	5.77	0.40	1.80	2.41	2.50	0.30
LiCl solution, 200	11.56	0.85	3.65	4.83	5.08	0.70
Coolant "LiCl", 400	23.13	1.72	7.33	9.67	10.16	1.42
LiCl solution, 600	30.12	2.29	10,29	14.49	15.24	2.10
NaCl solution, 100	2.32	0.08	0.23	0.94	1.00	0.14
NaCl solution, 200	4.76	0.21	0.46	1.89	2.02	0.30
NaCl solution, 400	9.54	0.42	0.92	3.78	4.03	0.63
NaCl solution, 600	14.24	0.72	1.41	5.24	6.01	0.82
NaCl solution of Slovyansk deposit, 408	9.52	0.48	1.03	3.92	4.20	0.56
NaCl solution of Drogobych deposit, 389	11.06	0.59	1.07	3.34	3.38	0.63

Note: * – averaged results of six experiments are presented. Ultrasound intensity is 20 W/cm^2, and the solution temperature is 20±1°C.

Table 2

Dependence of the sonoluminescence intensity of sodium, lithium, and cesium chlorides on the intensity of the initiating ultrasound

Sample and solution concentration, g/dm^3	Sonoluminescence intensity (arbitrary units) at different ultrasound intensity			
	17 W/cm^2	18 W/cm^2	19 W/cm^2	20 W/cm^2
Coolant "CsCl-1", 400	23.15	26.33	31.05	35.23
Coolant "CsCl-2", 600	36.03	40.76	46.50	52.84
Coolant "LiCl", 400	6.71	7.95	8.96	10.16
NaCl solution, 400	2.54	2.73	3.55	4.03
NaCl solution, 600	3.85	4.06	5.32	6.01
NaCl solution of Slovyansk deposit, 408	2.57	3.08	4.03	4.20
NaCl solution of Drogobych deposit, 389	2.79	3.21	3.23	3.38

20 W/cm^2.

The accuracy of the developed method was verified by comparison with gravimetric analysis and sonoluminescence spectroscopy using low-frequency and high-frequency ultrasound.

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Table 3

Results of determining the main component in brines and highly concentrated technological solutions (n=6; P=0.95)

Sample, concentration, g/dm^3	Addition, g/dm^3	Determined value, g/dm^3							
		By sonoluminescence method						By gravimetric method [6]	
		US 22.0 кHz		US 12.0 MHz		US 22.0 MHz		$\overline{C}$	S_r
		$\overline{C}$	S_r	$\overline{C}$	S_r	$\overline{C}$	S_r		
Coolant "LiCl", 400	–	381	0.042	392	0.032	398	0.011	397	0.011
	50	412	0.044	428	0.031	441	0.012	442	0.012
Coolant "CsCl-1", 400	–	381	0.042	391	0.026	392	0.011	394	0.011
	50	402	0.041	421	0.027	443	0.013	448	0.010
Coolant "CsCl-1", 400	–	571	0.042	572	0.037	591	0.012	594	0.011
	100	646	0.047	687	0.038	692	0.013	673	0.011
NaCl solution of Slovyansk deposit, 408	–	381	0.045	388	0.032	404	0.016	402	0.012
	50	412	0.044	423	0.033	448	0.017	446	0.012
NaCl solution of Slovyansk deposit, 408	–	364	0.051	375	0.035	381	0.017	381	0.013
	50	402	0.052	418	0.034	431	0.015	433	0.014
NaCl solution, 400	–	374	0.051	383	0.033	391	0.014	393	0.012
	50	402	0.053	421	0.033	442	0.015	442	0.013
NaCl solution, 400	–	558	0.051	572	0.037	589	0.016	591	0.011
	100	617	0.053	678	0.037	681	0.015	649	0.011

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СОНОЛЮМІНЕСЦЕНТНА СПЕКТРОСКОПІЯ З ІНІЦІАЛІЗАЦІЄЮ СОНОЛЮМІНЕСЦЕНЦІЇ НАДВИСОКОЧАСТОТНИМ УЛЬТРАЗВУКОМ: ВИЗНАЧЕННЯ ВМІСТУ ОСНОВНОЇ РЕЧОВИНИ У ВИСОКОКОНЦЕНТРОВАНИХ РОЗЧИНАХ

Т.В. Черножук

Продовжено дослідження нового методу аналізу «сонолюмінесцентна спектроскопія», на прикладі визначення вмісту основної речовини в природних розсолах і технологічних висококонцентрованих розчинах, з використанням для ініціалізації сонолюмінесценції ультразвуку надвисоких частот (≥10,0 МГц). Встановлено закономірності впливу частоти ініціюючого сонолюмінесценцію ультразвуку на інтенсивність сонолюмінесценції хлоридів цезію, літію та натрію. Доведено тотожність процесів утворення і схлопування кавітаційних пухирців для розчинів хлоридів лужних металів. Показано, що максимуми інтенсивності сонолюмінесценції для хлоридів лужних металів досягаються при частоті ультразвуку 20–22 МГц. Розроблено методику визначення вмісту цезію, літію і натрію у висококонцентрованих технологічних розчинах та розсолах з використанням ультразвуку надвисокої частоти для ініціалізації сонолюмінесценції. Правильність методики перевіряли аналізом одних і тих же проб альтернативними методами: гравіметрією, сонолюмінесцентною спектроскопією з використанням для ініціалізації сонолюмінесценції ультразвуку високої та ультразвуку низької частот.

Ключові слова: сонолюмінесценція, кавітаційні пухирці, висококонцентровані розчини, природні розсоли, ультразвук, відносне стандартне відхилення.

SONOLUMINESCENT SPECTROSCOPY WITH SONOLUMINESCENCE INITIATION BY ULTRAHIGH-FREQUENCY ULTRASOUND: DETECTING THE CONTENT OF THE MAIN SUBSTANCE IN HIGHLY SATURATED SOLUTIONS

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Keywords: sonoluminescence; cavitation bubbles; highly concentrated solutions; natural brines; ultrasound; relative standard deviation.

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