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Employment of Low Frequency Ultrasonic Waves Assisted with Silica Particles for the Separation of W/O Emulsions

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Abstract

Heavy fuel oil is a complicated amalgamation that generally include substantial quantities of salt water, leading to stable emulsions. As these fluids traverse pipelines and constricted valves, the water contained is emulsified, resulting in "water-in-oil" emulsions. These emulsions are stabilized by natural compounds found in heavy oil, including asphaltenes, resins, waxes, and solid particulates. Water contamination in heavy fuel oil is detrimental, resulting in various issues such as pipeline damage and elevated transportation expenses. Consequently, it is imperative to devise efficient water-oil separation systems, considering economic and environmental considerations. This study seeks to examine the impact of low-frequency ultrasound (20 kHz) on the demulsification of water-in-heavy fuel oil emulsions, while enhancing separation efficiency by the utilization of silica particles. Silica particles measuring smaller than 53 µm were utilized at several concentrations (5,000, 10,000, 15,000, 20,000, and 25,000) ppm to enhance separation efficiency. The impact of ultrasonic exposure length (3, 6, 9, 12, 15) minutes at 100 W power and 70°C was evaluated. The findings indicated that the optimal separation efficiency (85%) was attained using 20,000 ppm silica nanoparticle powder, utilizing a 15-minute exposure duration and 100 W power. The findings validate that the integration of ultrasound and silica nanoparticles constitutes an efficient, eco-friendly, and cost-effective method for the separation of water from heavy fuel oil emulsions.

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1. Introduction

A principal challenge in heavy oil production is the separation of water-in-oil (W/O) emulsions. Emulsified water not only increases the susceptibility of pipelines and equipment to corrosion [1], but also enhances the viscosity of heavy oil, resulting in energy loss during transportation. Demulsification can be achieved by many combinations of physical, chemical, or biological mechanisms, as is commonly acknowledged [2]. The heavy oil business extensively employs procedures such as demulsifier addition and electrostatic treatment. An appropriate demulsifier is often employed to facilitate the demulsification process. The major purpose of the chemical

additions is to destabilize the emulsifying agents [3]. Demulsifiers enhance droplet coalescence in emulsions by adhering to and destabilizing the oil-water interface [4]. Mechanical demulsification entails using the density disparities between the water and oil phases or disrupting physical barriers. Consequently, a proficient, economical, and environmentally benign demulsification technique is necessary [5]. Also, the amalgamation of heavy oil and salty water must undergo demulsification, or division into two distinct phases, before the heavy oil can be transported or processed [6].
A number of demulsification techniques, including membrane filtration, centrifugation, and microwave radiation, have been developed for the petroleum industry or for the laboratory scale [7], [8]. Even though these processes are fairly effective for light-to-heavy oils, they take a lot of time, and the inclusion of chemical demulsifiers is nearly always required (particularly for heavy crude oils), which raises treatment costs [9].

According to Saat et al. [10] studied the possibility of employing coconut oil and its byproducts, such as coconut betaine, as environmentally friendly demulsifiers to separate crude oil emulsion from water. In every trial examining the effect, the results indicated that coconut betaine was more beneficial than coconut oil. At 70 °C, 3 mL of coconut betaine in a 10 mL crude oil emulsion without the addition of xylene produced the maximum water separation of almost 35%. Liu et al. [11] investigated how various heavy oils' physical-chemical viscosity is reduced by ultrasonic waves. The degree of cavitation viscosity reduction, ultrasonic physical disturbance, and ideal ultrasonic parameters for several oil samples were ascertained. The findings indicate that the key to lowering heavy oil's viscosity is its initial viscosity, temperature, and water content. The first oil sample's higher viscosity, higher water content, and higher temperature were required. Hassan et al. [12] studied the technical and An economic analysis of the application of silica nanoparticles to improve crude oil demulsification. For the Central Processing Facility (CPF), which processes heavy crude oil from Fula in Balila, Sudan, an MS Excel software model was created. The primary production cost and Net Present Value (NPV) characteristics were subjected to a sensitivity analysis for several flow schedule selection choices. The production cost is reduced by 19% when comparing flow tables based on identical plant capacity, but only 3.7% when comparing based on constant yearly crude oil processing. Azubike st al. [13] presented an evaluation of the potential of breaking crude oil emulsions using demulsifiers produced from tobacco seed oil, leaf extracts, and stem ash. Both hydrophilic and hydrophobic demulsifier components were found in the physiochemical characteristics of Nicotiana tabacum seed oil, the phytochemical analysis of Nicotiana tabacum leaf extracts, and the potash concentrations of Nicotiana tabacum stalk ash extract. The results of the bottle tests demonstrated that the demulsifier's formulation can shatter medium- and high-density crude oil emulsions. Ye et al. [14] employed a straightforward hydrothermal technique to extract emulsions from crude oil emulsions using natural lotus leaves. The HLLF powder's unique wettability and surface structure have demonstrated its efficacy in breaking W/O emulsions quickly. When the HLLF dosage was 1000 mg/L and the settling duration was 90 minutes at 70 °C, DE could reach 88.17%. Additionally, HLLF's demulsification capabilities were comparable to those of a few widely used commercial demulsifiers. Additionally, it could successfully demulsify two different kinds of crude oil emulsions from various oilfields. Yuan et al [15] synthesized an eco-friendly RHC-0.5 demulsifier through a straightforward one-step hydrothermal process utilizing rice husk (RH) as the primary material. Characterization was conducted using Fourier transform infrared spectroscopy, X-ray photoelectron spectroscopy, field-emission scanning electron microscopy, energy dispersive X-ray spectrometry, and thermogravimetric analysis. An optimal DP of 95.01% was achieved with 300 mg/L of RHC-0.5 at 70 °C for 10 minutes. Furthermore, RHC-0.5 demonstrated commendable salt tolerance, even at a salinity of 40,000 mg/L. Additionally, its DP was compared with that of commercial demulsifiers such as SP169, CH-66, and AR-32. Parvasi et al. [16] demonstrated the effects of low-frequency ultrasound on the demulsification of low crude oil content (500 mg/L) from a crude oil wastewater unit. subsequently conducted studies using a laboratory ultrasonic bath at frequencies of 25 kHz and 45 kHz without the use of chemical demulsifiers. Create experiments to assess how wastewater temperature, ultrasound power, ultrasound frequency, and ultrasound irradiation duration affect separation efficiency. The findings showed that the efficiency of crude oil emulsion separation rose by 72% when ideal circumstances were met. Nadirov et al. [17] conducted a comparative study of the demulsifying ability with respect to no emulsifier for original and freshly milled quartz (FMQ) particles isolated from river sand. In addition to standard solid-liquid characterisation techniques, use rheological measurements, surface tension measurements, and demulsification tests to observe how quartz with a 75-micron mesh size reduces emulsion stability. After 100 minutes of deposition, a demulsification efficiency of 97% was attained with the addition of 3 weight percent FMQ, as opposed to 140 minutes for pristine quartz. The purpose of grinding quartz is to make water more admeable, which raises the pH level locally and causes the emulsion to become less stable and eventually dissolve. Okereke et al. [18] intends to use locally produced raw ingredients to design and formulate an inexpensive, ecologically friendly demulsifier. To determine how well it broke the emulsion of crude oil, an experimental examination was conducted. Castor oil, distilled water, starch, camphor, alum, and locally produced liquid soap were among the materials utilized. A crude oil emulsion sample from a Niger Delta field was used to evaluate two distinct demulsifier formulations, which were then heated to 60°F. The treatment's outcome was the effective use of a demulsifier to separate the oil from the water. Gao et al. [19] discovered Microscopic analysis, particle size analysis, component analysis, and other techniques are used to determine the microscopic topography, particle size, components, etc. of oil samples before and after ultrasonic irradiation. The findings demonstrate that, in the presence of ultrasonic cavitation, the oil samples accomplish demulsification and dehydration, with a maximum dehydration rate of 98.24%. The outcomes of particle size and microscopic investigations show that ultrasonic irradiation destabilizes the oil-water interfacial barrier and leads to the collision, agglomeration, and settlement of droplets of various sizes. Additionally, the emulsion system's droplets were found to be more uniformly dispersed and to have larger intervals. Mirzadeh et al. [20] investigated the factors affecting the variables of the two-part demulsifier formulation.First, using the hydrophilic-lipophilic (HLD) deviation concept, the effectiveness of five demulsifier combinations with the same surfactant coefficient was examined. Additionally, the impact of adding alcohol (c-butanol) on water separation in crude oil emulsions was tested. The demulsifier mixture of lauryl-myristyl-alcohol-3ethoxylate (KELA3) and sodium dodecyl sulfate (SDS) had the highest water separation efficiency, with 75%, according to the data. For surfactant and oil-water systems with various salts, such as the demulsification performance of heavy crude oil emulsions, the second section sought to assess the prediction efficacy of the HLD-NAC (network average curvature) model. Li et al. [21] developed a dynamic self-releasing membrane that generates a high concentration of hydroxide ions on its surface, resulting in interfacial demulsification and antifouling properties. By markedly diminishing their Debye length and obstructing their electric double layer repulsion, these hydroxide ions facilitate the demulsification of emulsified oil droplets on the membrane surface. The issues of membrane fouling and trade-offs may be mitigated if the emulsified oil droplets coalesced into larger aggregates and rapidly detached from the membrane surface. In the long-term separation of oil/water emulsions, the membrane exhibits remarkable antifouling properties, achieving an efficiency of 99.79% and a flow of 3046 L m^{-2} h^{-1} bar^{-1}. The aim of the present work is to develop an eco- friendly technique presented by using of low frequency ultrasonic wave to suppress the emulsion assisted with low cost and locally produced silica particles. The impact of operating parameters is a crucial issue in ultrasonication demulsification. The research contributes separation efficiency by advancing more effective methods in petroleum processing, paving the path for future investigations into additional elements that might beneficially influence this process. Therefore, the study also investigates the effect of sonication time and power in addition to silica concentration on W/O emulsion separation.

2. Experimental methods:

2.1 Materials:

The heavy fuel oil emulsion equipment used in this research was obtained from the Daura refinery in Baghdad, Iraq, which was used to make water-in-oil emulsions. Several samples were prepared in (100 ml) glass beakers containing (60% oil and (40%) water and a specific volume of (5500 ppm) sodium chloride solution was mixed to prepare the emulsions [22]. Table1 displays this oil's physical characteristics.

Table 1: Physical properties of the Basra Heavy oil. [Al-Doura refinery]

Heavy fuel oil properties	
sp. gr at 15.6 ºC	0.8724
API at 15.6 ºC	15.8
Density at 15 ºC (g/Cm^3)	0.9657
Kinematc Viscosity at 50ºC cst (mm^2/s).	183
Pour point ºC	+3
Carbon residue %w/w	8.1
Sulfur content %w/w	4.5
Water & sediment %v/v	<0.025
Asphaltenes content (wt %)	3.09%
Calorific Value (gross) Keal/kg	10441

The chemical used in the separation process is silica with a particle size of less than (53 microns). The most effective emulsion bottle test method was used to break the emulsion. The test also indicates the minimum amount of solvent required to penetrate the experimental device [23].

The ultrasonic technology (PLS-XCSB300) is Lab Scale Ultrasonic Cell Crusher Ultrasonic Homogenizer 10mm 15mm Diameter Probe Sonicator. Lab ultrasonic homogenizer is also called ultrasonic cell crusher,ultrasonic probe sonicator,ultrasonic vibrating rod. The ultrasonic vibrating rod is mainly composed of ultrasonic transducer, ultrasonic horn and ultrasonic generator. It can be used in emulsification, separation, dispersion, homogenization, extraction, degassing, cleaning and accelerating chemical reactions, etc [24]. The specifications of the ultrasonic machine used in the experimental work are presented in Table 2.

Table 2: specifications of the ultrasonic machine

Specifications	Value
Type	PLS-XCSB100
Input voltage	220V 50-60 Hz
Frequency	19-20 kHz
Rated power	100 W
Working ambient temperature	+5 to +50℃
Tool head material	Titanium alloy
Length of the tool head	182 mm
Maximum operating temperature	150 degrees
Power supply size	380×285×135 mm
Connecting length	5 m
Reference processing capacity	1000 ml

Many devices were used for the experimental procedure, including: Digital Weigh balance to measure the weight of the added material, Borosilicate Glass 500 ml conical separatory funnel, Funnel, Flask conical to empty the emulsion after separation, Ring stand, Beaker, Magnetic Heat-Stirring machine to mix water with oil, Glass rod to mix the chemical with the emulsion, Ultrasonic device and Stop watch.

2.2 Experimental procedure:

Figure (1) shows the process of applying ultrasound waves to petroleum emulsions in a 300 ml Beaker. They are then emptied into borosilicate Glass 500 ml conical separatory funnel containing a controlled tap at the bottom to empty the water content after separation into a 500 ml conical flask.

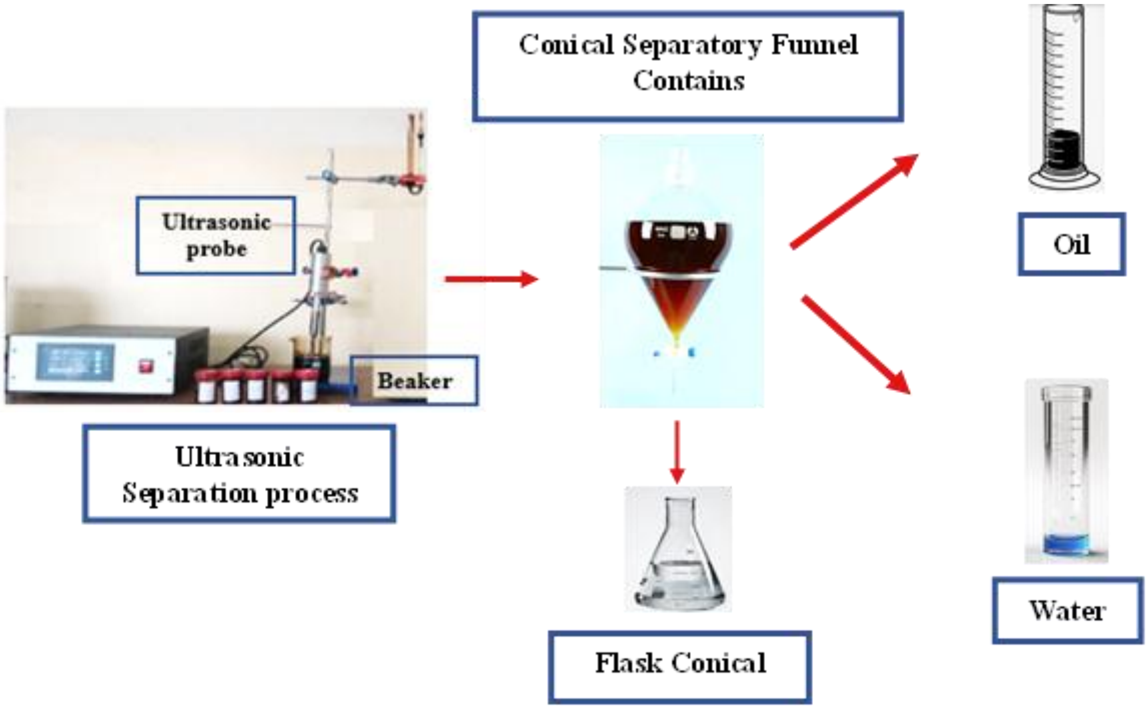

Fig. 1: Diagram of emulsion separation process.

After ultrasonic device mapping, W/O emulsion containing 40% of water was subject to sonication for the evaluation of demulsification efficiency. The experiments were performed at driving frequency of 20kHz and temperature of 70 °C With the addition of (20000 ppm) of silica chemical to the emulsion. The results obtained after the treatment of the heavy fuel oil emulsion in different regions of ultrasonic device inside of the conic glass reactors, are presented in (Fig. 2).

It can be seen in Figure 2 that a higher demulsification efficiency (from 75 to 82%) was obtained after adding the chemical over the converter. However, time and energy also play an important role in the separation process. Where notice that when the emulsion treatment period increases, the temperature rises due to friction, which facilitates the separation process. However, when the energy increases, the separation process is much faster.

To ensure the stability of the emulsion, the vessels holding the oil/water emulsion were left for 24 hours prior to separation before each experiment. The materials are exposed to ultrasonography, allowed to sit for five minutes, and then a reading is obtained. By considering the initial water content in the emulsion before ultrasound (IWC) and the final water content obtained after ultrasound exposure (FWC), the demulsification efficiency (ED) was calculated for all experiments in which the water-emulsion separation was visually observed. This was done using the following equation:

$$ED\% = \frac{I_{WC}-F_{WC}}{I_{WC}} \times 100 \ \ldots\ldots \text{ Eq (1). [25]}$$

Where:

I_{WC}: The emulsion's initial water content prior to ultrasonography.

F_{WC}: The ultimate water content measured during ultrasonic exposure.

Cannon-Fenske-Routine Viscometer was used to determine the kinematic viscosity of heavy oil. The relationship applied to calculate the kinematic viscosity is

$$\nu = K \times t \ldots \qquad \text{Eq (2).}$$

The instrument constant k refers to the timing marks during the visual survey of the meniscus passage.

It comes to: k=0.02688 mm^2/S^2.

3. Results and Discussion:

3.1 The relationship between emulsifier concentration and the efficiency of oil-water separation:

Chemical demulsification is the process of utilizing chemicals to remove water from oil. This process employs silica-based chemicals known as "demulsifiers" to extract water from oil. The use of silica facilitates molecular adhesion, hence expediting the separation process. The data indicate that the introduction of 5000 ppm of the chemical results in a separation efficiency of 70%. Conversely, an addition of 20000 ppm yields a separation percentage of 83%, which is considered optimal. This suggests that while increasing the dosage enhances separation, the improvement is marginal, as illustrated in Figure 2. Silica improves separation by augmenting the interaction between molecules, such as water and oil, near the surface. The surface may attain a saturation threshold when all surface sites have been occupied by the chemicals if silica is constantly introduced. Introducing more silica beyond this juncture may not influence the separation further, leading to a consistent separation efficiency. Silica functions as a demulsifier by altering surface tension and enhancing repulsion among water droplets, so aiding the separation process. Compared to Liu study [11].

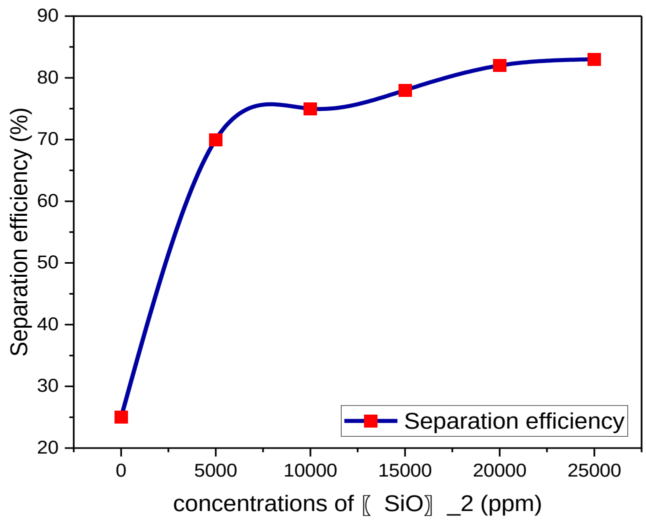

Fig. 2: Effects of water separation using various concentrations of SiO_2.

3.2 Effect of temperature on oil viscosity:

A mixture of hydrocarbons, heavy oil is difficult to flow because it becomes viscous at low temperatures. As seen in Figure (3), raising the temperature causes the oil's viscosity to decrease. Heat causes the oil molecules to move more quickly and weakens the molecular connections that prevent flow. The molecules may more easily slide past one another as the temperature rises, which lowers the viscosity and facilitates the separation of the water and oil. At elevated temperatures, interactions may transpire between the oil and water in the sample, resulting in the creation of more stable emulsions, hence augmenting viscosity. At elevated temperatures, certain heavy molecules in the oil may undergo decomposition, resulting in the formation of new substances with increased viscosity. Certain compounds may commence interactions, altering the molecular structure and enhancing viscosity. In accordance with Gandomkar's source [26].

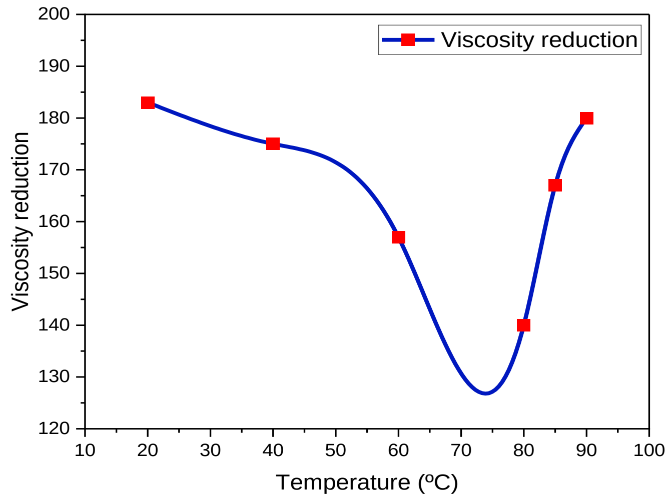

Fig. 3: The impact of temperature on the lowering of ultrasonic viscosity

3.3 Effect of temperature on separation efficiency:

At moderate temperatures, separation efficiency will generally improve due to reduced viscosity and improved oil flow, which helps speed up the separation process. Water and oil molecules travel more quickly, and molecular motion also accelerates with temperature. As seen in Figure (4), at low temperatures (about 20°C), the separation efficiency is suboptimal (around 10-20%). A marginal rise occurs between 20°C and 40°C; nevertheless, it remains minimal, suggesting that temperature alone is unable to disrupt the emulsion at this point. As the temperature ascends to around 50-60°C, the separation efficiency markedly improves, escalating from roughly 30% to beyond 70%. This suggests that warmth significantly contributes to the destabilization of the emulsion, facilitating water-oil separation. Post 60°C, the separation efficiency commences stabilization at about 75-80%. At 70-80°C, separation peaks at approximately 85%, signifying that the system has attained ideal thermal equilibrium, when separation efficiency is maximized but does not much enhance with additional temperature increments. an excessive temperature can have negative effects, cause more stable emulsions, or cause undesired interactions between chemical substances, all of which can make the separation process more challenging. The force that keeps water and oil from separating is called surface tension, and it is also influenced by temperature. In accordance with Hassan source [23].

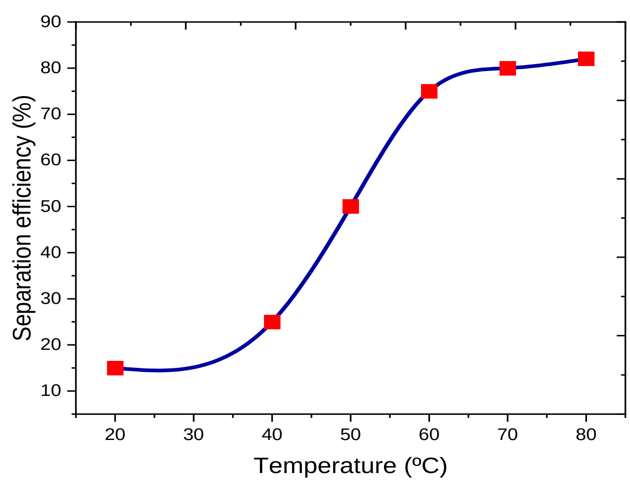

Fig. 4: Effect of temperature on separation efficiency.

3.4 Effect of time on separation efficiency:

Increasing the exposure time to ultrasound will break up the emulsified water molecules (dissolved in the oil) and form larger water droplets when adding an amount of (20000 mg/l) of silica as shown in Figure (5). The graph indicates that separation efficiency improves over time, beginning at around 63% at 3 minutes and attaining approximately 85% at 15 minutes. The interval of 3 to 9 minutes demonstrates a progressive enhancement in efficiency from 63% to 75%, signifying the initiation of silica particles' activation in the adsorption or contact process with heavy oil. The interval of 9 to 12 minutes signifies a considerable enhancement in efficiency, suggesting that the silica particles have attained their peak activity and exhibit great effectiveness throughout this duration. After 12 minutes, the improvement in efficiency becomes marginal, signifying that the system is nearing equilibrium or silica particle saturation, at which point it can no longer effectively separate more water. In accordance with Hassan source [23].

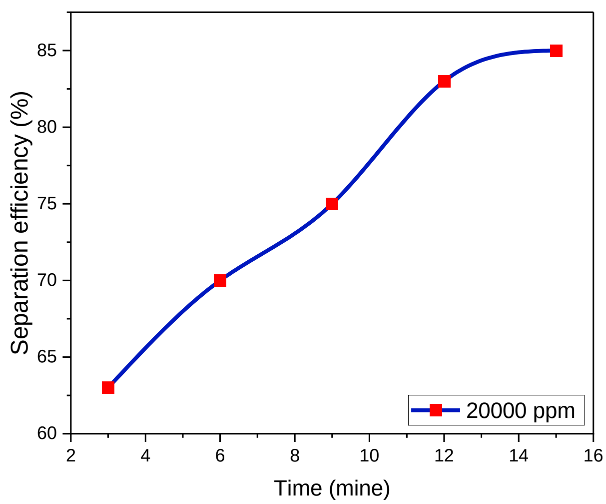

Fig. 5: Effect of time on separation efficiency.

4. Conclusions

The findings demonstrate that employing low-frequency ultrasound in conjunction with silica is an efficient method for demulsifying water-in-heavy fuel oil emulsions. This approach achieved a separation rate of 82% in 15 minutes at 100 W, illustrating its efficacy in enhancing separation processes. Minimizing emulsions in heavy fuel oil is essential for mitigating operational issues, including corrosion and elevated transportation expenses. This study's technique offers a cost-effective and eco-friendly alternative to traditional procedures, rendering it a viable choice for extensive industrial applications. Further research and development might refine this technology to accommodate diverse operating situations and improve the efficiency of emulsion treatment in the petroleum sector.

Abbreviation table:

Symbol	Definition
CFD	Central Processing Facility
NPV	Net Present Value
HLLF	Hydrophobic Lotus Leaf Functionalized
DE	Demulsification Efficiency
RHC	Rice Husk Carbon
DP	Demulsification Performance
FMQ	Freshly Milled Quartz
HLD	Hydrophilic-Lipophilic Deviation
KELA3	Lauryl-Myristyl-Alcohol-3Ethoxylate
SDS	Sodium Dodecyl Sulfate
NAC	Network Average Curvature
API	American Petroleum Institute
IWC	Initial Water Content
FWC	Final Water Content
ν	The kinematic viscosity mm^2/s
K	The instrument constant is 0.02688 mm^2/S^2
t	Time
μm	Micro meter
Sio_2	Silicon dioxide

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