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Synthesis, Characterization, and Evaluation of an Eco-friendly Demulsifier for Crude Oil Emulsion Treatment Using Waste Corn Oil

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P A P E R I N F O

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A B S T R A C T

Despite the diversity of energy sources at the present time, they have not been able to be a real alternative to crude oil, as it is still considered the primary source of energy in the world and will remain so for many years. As is known, the petroleum industry consists of an interconnected series of operations, starting with extracting crude oil from wells and ending with its refining process. As is known, the petroleum industry consists of an interconnected series of operations, starting with extracting crude oil from wells and ending with its refining process. These operations vary in degree of difficulty, cost, and challenges they face. Crude oil emulsion is one of the most costly issues in this important industry. In the current study, a novel environmentally friendly bio-demulsifier synthesized from corn oil waste has been introduced. The unique characteristics of this novel bio-demulsifier were diagnosed using several tests, including: Fourier transform infrared spectroscopy (FTIR), gas chromatography-tandem mass spectrometry (GC-MS), proton nuclear magnetic resonance (1HNMR), and thermogravimetric analysis (TGA). The demulsification activity has been evaluated using a bottle test method as a function of settling time, water content, and temperature. The maximum separation ratio was 69.3% at a dose of 1000 ppm for 5 hours and 70 °C, while the water-oil ratio was 30/70. The obtained results demonstrate that bio-demulsifier could be used as a safe and environmentally friendly alternative in initial demulsification units, which will reduce the environmental hazards and financial costs associated with the oil industry when using traditional demulsification methods.

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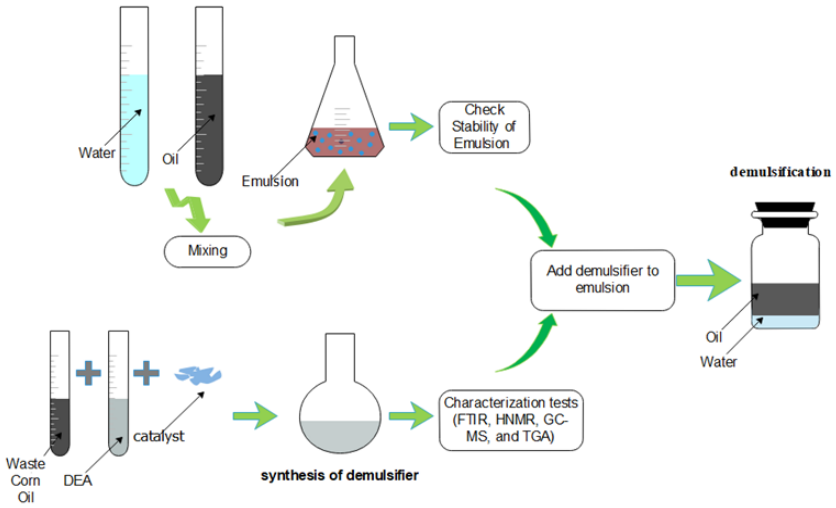
Graphical Abstract



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1. INTRODUCTION

The oil industry is still the largest supplier of the global energy sector, and the rest of the energy industries have not risen to compete with this industry despite making great strides in development and growth. Reaching the stage of serious competition between renewable energy sources and those provided by the oil industry will take a long time. Therefore, it is necessary to continue developing the oil industrial sector and make it as safe and environmentally friendly as possible (1, 2). In order to achieve the aim of sustainability in the oil industry, attention must be paid to the issue of chemicals that enter the process of preparing crude oil emulsions. Crude oil emulsions can be defined as systems with two or more immiscible liquids distributed as droplets with the assistance of an emulsifying agent (3). The continuous phase surrounds the droplets, which represent the internal phase. The emulsifying agent stabilizes the emulsion and separates the dispersed droplets from the continuous phase (4, 5). The most common types of crude oil emulsions found in petroleum production are oil-in-water emulsions (O/W), water-in-oil emulsions (W/O), and more complex water-in-oil emulsions (W/O/W) (4, 6). Water-in-oil emulsions affect oil production and processing. Hydrocarbon reserves, production, refineries, and pipelines can generate emulsions (4, 7). Crude oil may include water droplets for many reasons. Due to its composition, crude oil contains less than 50 μm water droplets. Drilling causes turbulent mixing and agitation that disperse water droplets in the oil system (8, 9). Since water is considered one of the causes of corrosion in oil pipelines crude oil viscosity, and catalyst poisoning during refining (10-12). Therefore, it requires the removal of emulsified water during the process of transporting and refining crude oil. The process by which dispersed water is separated from the crude oil phase is called demulsification (13). Different techniques, such as electrical, thermal, membrane filtration, microwave, and physical/chemical methods, can be applied to process oil emulsions (14-16). Chemical demulsifiers/heating are the most efficient approaches to water dehydration. Demulsifiers work by diffusing chemicals into emulsions, replacing the asphaltene layer around water droplets with a soft film that encourages droplet coalescence, and allowing gravity to separate emulsified water (4, 14). Fermentation, transesterification, and methanation are examples of biotechnology procedures that may be used efficiently in waste biorefineries that use waste oil. Industrial production of biodiesel and biogas from waste oil can create value-added products (17). Biosurfactants have shown superior performance compared to chemical surfactants, where they exhibit high stability at higher pH and temperature. The most critical parameter is that these materials are biodegradable, biocompatible, and less toxic. Compared to conventional surfactants, biosurfactants can recover more oil, and they are both economical and ecologically friendly (18, 19). Commercial oils like maize oil (20), castor oil (21, 22), linseed oil, and soybean oil (23) can be used to make demulsifiers. Waste corn oil, a popular vegetable oil that is inexpensive, non-toxic, and abundantly renewable, has promise as a suitable material. The ultimate aim of this study is to develop a novel, eco-friendly, and cost-effective demulsifier derived from corn waste oil for separating water-in-oil emulsions (W/O). FTIR, GC-MS, TGA, and 1HNMR methods will be used to characterize the produced surfactant. The bottle test technique will be used to evaluate its demulsification effectiveness as a biodemulsifier.

2. MATERIALS and METHOD

2. 1. Materials The materials used in this study included the following:

1. Waste corn oil for frying: This material was obtained from local vegetable oil factories.
2. Diethanolamine (DEA): has a purity of 98%. DEA was supplied by Thomas Baker, India.
3. P-Toluensulphonic Acid Monohydrate: with a purity of 98.5%, is supplied by HIMEDIA, India.
4. Sodium chloride: is supplied by Alpha Chemika, India, with 99.5% purity.
5. Ether Petroleum, 40–60 °C: with 98% purity. This material was supplied by Alpha Chemika, India.
6. Sour crude oil: The sour crude oil sample was taken from Al-Dura refinery, which was extracted from Basrah crude oil in Iraq. The properties of this sample are listed in Table 1.

2. 2. Demulsifier Synthesis The demulsifier preparation process consists of four steps, which procedures have been completed according to the reference (20):

1. First step: waste corn oil has been filtered to eliminate impurities and solid carbonous particles.
2. Second step: involved reacting waste corn oil with diethanolamine at a ratio of 2:1 (v%) using the base catalyst p-toluene sulfonic acid monohydrate. The reaction occurred in a 500 mL flask fitted with a reflux

TABLE 1. Physical properties of sour crude oil

Property	Value
Sp.Gr. at 15.6°C	0.88490
API	28.40
Viscosity (cp) at 20 ℃	19.4
Sediment and Water content (%vol.)	0.050
Asphaltenes (%wt.)	2.220

condenser, thermometer, and magnetic stirrer. Initially, the waste corn oil (66.6 mL) and catalyst (0.83 g) were heated to 140 °C while stirring until the catalyst dissolved, which took approximately 1 hour. Diethanolamine (33.3 mL) was gradually added, and the temperature was increased to 180 °C for one hour. The reaction was then allowed to proceed for an additional 2 hours with continuous stirring, during which water (8 mL) was collected as an indicator of the chemical reaction. The resulting product was collected and allowed to cool to room temperature.

3. Third step: an excess of petroleum ether was added to the product and mixed thoroughly for 20 minutes.

4. Fourth step: involved purification, where the solvent, along with contaminants and unreacted materials, was removed using the evaporation technique via a rotary evaporator under vacuum conditions at a temperature of 50 °C and a rotation speed of 100 rpm.

2. 3. Emulsion Preparation In order to create conditions similar to those found in the crude oil industry, a simulated crude oil emulsion was prepared by mixing a brine solution with a 3% concentration (0.5M NaCl solution) with crude oil in a volumetric ratio of 30/70. The brine solution was chosen as an imitation of the aqueous solution commonly employed in the field of crude oil. The crude oil emulsion was homogenized at 10000 rpm for 30 minutes at ambient temperature. The stability of the emulsion was evaluated through visual observation and the analysis of droplet size distribution using an optical microscope.

2. 4. Demulsification Experiments The bottle test was performed to evaluate the demulsifier's performance. The demulsifier was added in various amounts, ranging from 1000 ppm to 6000 ppm, to each 100 mL of the emulsion. Using a graduated measuring container and the measured volume of water in milliliters, Equation 1 was used to compute the water separation efficiency (WSE%):

$$\text{WSE } \% = \frac{\text{volume of separated water (ml)}}{\text{Total volume of water in the sample (ml)}} * 100\% \qquad (1)$$

The graph illustrates three distinct dispersion rates. In addition, the different parameters were determined to reach the optimum conditions.

3 .RESULTS AND DISCUSSION

3. 1. structure and Bonding Characterization

3. 1. 1. Fourier Transform Infrared Spectroscopy (FTIR) This test was done to detect the bonding of the demulsifier.The range of wavelength of FTIR spectra was between 400 and 4000 cm^{-1} using liquid cells. The test was done via FTIR type 8400S manufactured by Shimadzu/ Japan). This test was done to detect the bondings of the demulsifier.

The FTIR spectra of the synthesized demulsifier (WMF) in Figure 1 show a broad peak at 3375 cm^{-1} sequentially, which, according to its classification as secondary aliphatic alcohols, indicates O-H stretching. The two stretching peaks of NH_2 disappearance at 3375cm^{-1} had confirmed interactions of hydroxyl groups with amine groups (24).

TIR spectra show NH_2 stretching peaks at 3100-3400 cm^{-1}. These peaks represent amine group N-H stretching vibrations. N-H stretching peaks may shift, expand, or disappear due to amine-hydroxyl group interactions. Hydrogen bonds between hydroxyl and amine groups weaken the N-H bond, lowering its vibrational frequency and shifting or eliminating the peak (24).

Sharp absorption peaks at 3008–2855 cm^{-1} are given to both symmetrical and asymmetrical alkane stretching ($–CH_3$) and alkenes ($=CH_2$) in the aliphatic hydrocarbon groups (20).

The peaks at 1739 and 1165 cm^{-1} refer to the presence of fatty acids (C=O) stretching of esters. This is clear evidence of ester formation (25). The band at 1053 cm-1 is the major group of aliphatic alcohols (C-OH) (20). As illustrated in Figure 1, several functional groups in surfactants, such as hydroxyl, carboxyl, fats, aliphatic alcoholic, and alkane compounds, were found by the examination of the FTIR spectrum data.

3. 1. 2. Gas Chromatography-mass Spectrometry (GC-MS) Gas chromatography-mass spectrometry (GC-MS) is a powerful analytical technique used to identify and quantify chemical components in complex mixtures. The GC can separate compounds in a mixture according to their physical qualities, whereas the mass spectrometer can identify them based on their structure and molecular weight (14). GC-MS has the ability to

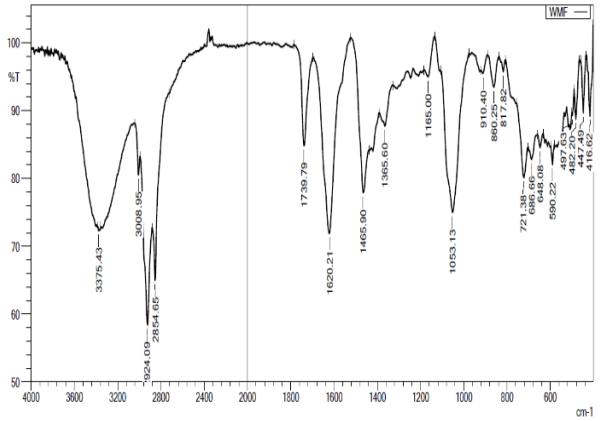

Figure 1. The demulsifier FTIR

detect and quantify a vast variety of chemical substances, even at very low concentrations, making it a valuable tool for the characterization of the demulsifier.

As shown in Table 3, the analysis of the GC mass results reveals that the demulsifier is a complex mixture of various compounds. The most abundant compound found in the demulsifier is 1,4-Bis(2-Hydroxyethyl)-Piperazine (BHEP), with an area percentage of 24.77%. BHEP has properties that make it effective in breaking emulsions and aiding in the separation process. In general, hydroxyethyl piperazine is intermediate in the manufacture of corrosion inhibitors, polyurethane catalysts, synthetic fibers, surfactants, and pharmaceutical.

Phenyl-3-methylpenta-1,2,4-triene with an area percentage of 17.47 %, also known as isopropenylbenzene or alpha-methylstyrene. It is an aromatic compound that belongs to the class of styrenes and could have a positive effect. It is widely believed that demulsifiers containing aromatic groups exhibit exceptional speed and performance. This can be attributed to the similarity between the aromatic groups in demulsifiers and those found in asphaltenes, which are known to generate the least stable oil film. Therefore, it is advantageous to have aromatic compounds in demulsification breakers, as an example methsuximide, flupentixol, 2-Cyclopropen-1-one, 2-cyclopropyl-3-(dichlorocyclopropyl methyl)-(CAS), and 5,8-dibromo-2-phenyl-1-aza-azulene, as they may improve the efficiency and effectiveness of the demulsification process (26).

On the other hand, there is a group of different compounds that have been found to assist in the process of breaking emulsions due to the existence of hydroxyl and amine groups (27), such as Ethanol, 2-[(2-aminoethyl) amino]-, and ribitol. Due to the absence of hydroxyl and amine groups, the remaining components are thought to have no influence on demulsification.

TABLE 2. Technical requirements for GC/MS operation

Gas Chromatograph	
Analytical Column	Agilent HP 5 MS/USA (30 m x 0.25 mm x 0.25 µm)
Injector-Port	Split (80:1)
GC Inlet Temp	260℃
Carrier-Gas	Helium
Injection Volume	1µl
Oven Program	
Temperature	60 ℃
Hold Time	4 min
Rate	3 ℃
Temperature	100 ℃
Rate	4 ℃
Temperature	260 ℃
Mass Spectrometer	
Auxiliary heat	280 ℃
Mass Variation via Full Scan Method	50-500

TABLE 3. Surfactant chemical composition as identified by GC-MS

No.	RT (min)	Name	chemical formula	Area (Ab*s)	Area%	CAS Number
1	9.083	Ethanol, 2-[(2-aminoethyl) amino]-	$C_4H_{12}N_2O$	409458786	3.73	000111-41-1
2	10.033	Ribitol	$C_5H_{12}O_5$	442973316	4.04	000488-81-3
3	17.702	1,4-BIS(2-HYDROXYETHYL)-PIPERAZINE	$C_8H_{18}N_2O_2$	2716336661	24.77	000122-96-3
4	22.615	Methsuximide	$C_{12}H_{13}NO_2$	724236388	6.60	000077-41-8
5	27.638	Linoleic acid, methyl ester	$C_{18}H_{32}O_2$,	295894561	2.70	000112-63-0
6	29.174	Flupentixol	$C_{23}H_{25}F_3N_2OS$	561733856	6.12	002709-56-0
7	34.861	Methimazole	$C_4H_6N_2S$	635394394	5.79	000060-56-0
8	36.915	Heptanoic acid, 7-bromo-, methyl ester	$C_{11}H_{21}BrO_2$	514522050	4.69	054049-24-0
9	37.061	Methyl 9,10-dichlorooctadecanoate	$C_{19}H_{36}Cl_2O_2$	512381414	4.67	033094-27-8
10	40.236	2-Cyclopropen-1-one, 2-cyclopropyl-3-(dichlorocyclopropylmethyl)- (CAS)	$C_{20}H_{27}Cl_2NO_3S$	374996861	3.42	062688-80-6
11	42.317	5,8-dibromo-2-phenyl-1-aza-azulene	$C_{15}H_9Br_2N$	353097888	3.22	121505-50-8
12	42.654	1-phenyl-3-methylpenta-1,2,4-triene	$C_{12}H_{12}$	1917606567	17.47	093247-38-2

3. 1. 3. ^{1}H NMR Spectroscopy The ^{1}HNMR (Proton Nuclear Magnetic Resonance) test looks at how hydrogen atoms move in a magnetic field to figure out the shape and chemical surroundings of molecules. It offers valuable insights into molecular connectivity, functional groups, and stereochemistry and finds applications in fields like chemistry, pharmaceuticals, and biochemistry. In this study, 1H nuclear magnetic resonance spectroscopy (^{1}H NMR) using a BioSpin GmbH Bruker spectrometer/ Germany was performed to verify the WMF synthesized chemical structure.

The compound's structure was assessed using ^{1}H NMR. The chemical shifts, multiplicities, coupling constants, and correlations of the NMR peaks, together with any other relevant information, were accurately measured.

Figure 2 shows the demulsifier structure. on the basis of the chemical components ^{1}H NMR (DMSO, 500 MHz) spectra, Demulsifier spectra show amine groups (NH_2 and NH) as a single peak and double peak at 0.84-1.5 ppm (28). At the same time, the (CH_2)n peaks of the alkyl group show up many times at 2.01 ppm and the -OH proton of WMF at 2.52 ppm (9). The chemical shift value of 3.46-5.35 typically indicates the presence of a proton signal in the region of aliphatic protons. It is less commonly associated with aromatic protons, as they typically appear in a higher chemical shift range (28).

3. 1. 4. Thermal Gravimetric Analysis (TGA) A thermogravimetric analyzer (TGA) from TA Instruments (SDT Q600 model/ USA) was used to determine the thermal stability of the synthesized demulsifier. TGA measures the amount of demulsifier mass lost when the temperature changes. The test was conducted in an Argon environment, with temperatures ranging from 25 to 800 degrees Celsius and a heating rate of 20 degrees Celsius/min.

Figure 3 presents the results of a thermal stability investigation of the WMF demulsifier. There were two stages of mass reduction. The first zone relates to the loss of weakly linked water molecules and has a mass loss of 7.584% from room temperature to 150 °C .

The second zone, from 150 to 800 °C, saw a dramatic decrease in mass (89.73%), indicating that at temperatures above 150 °C, WMF molecules begin to decompose. At 800 °C, the ash content is about 3%, and this indicates that the majority of the compounds in WMF are light ones.

The average temperature of a reservoir is between 50 and 120 degrees Celsius (13). WMF thermal stability is good because the water content makes it lose less than 5% of its weight. This can be fixed by adding a drying step at the end of the manufacturing process.

3. 2. Emulsion Stability The droplet size distribution of the simulated emulsion was characterized using an optical microscope (BVH340, Altay Scientific, Belgium)

The stability of the emulsion is a very crucial factor specified in the demulsification process. In this study, two approaches were used to specify the stability of simulated crude oil emulsions. The first one is visual observation. The duration observation continued for 72 hours after the synthesis process, and the separation ratio was recorded with time.

The second technique involved using an optical microscope to measure the droplet sizes of a drop-off generated emulsion (Figure 4 depicts the average droplet size and droplet size dispersion).

According to the emulsion stability determined by optical microscope methods, the emulsion can be classified according to the mean diameter. In this study, the mean diameter was 27.15μm, which means that the emulsion is stable, where the range of macro-emulsions is (0.5-100 μm) (29).

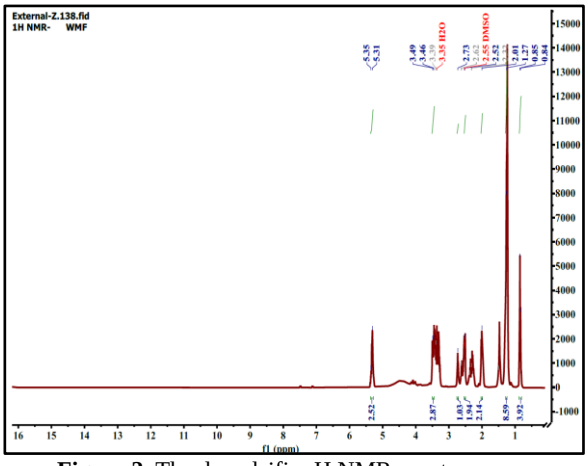

Figure 2. The demulsifier H NMR spectroscopy

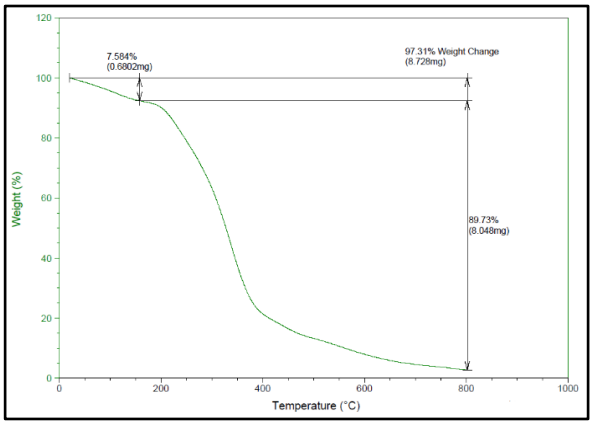

Figure 3. The demulsifier Thermal Gravimetric Analysis

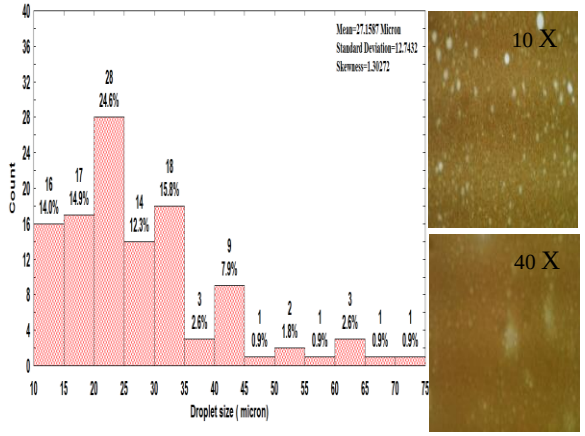

Figure 4. Images of the droplet dispersion in an emulsion, captured using a microscope.

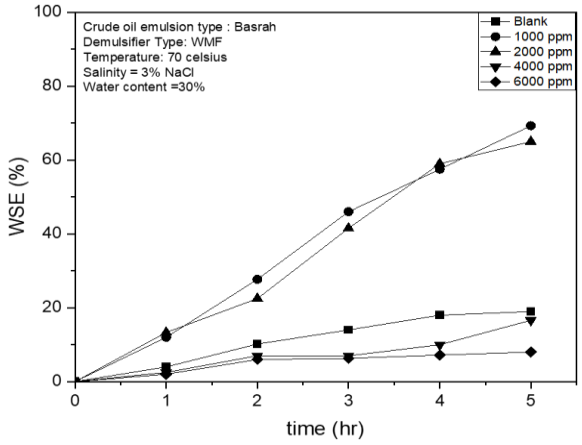

Figure 5. Demulsifier doses and emulsion separation efficiency

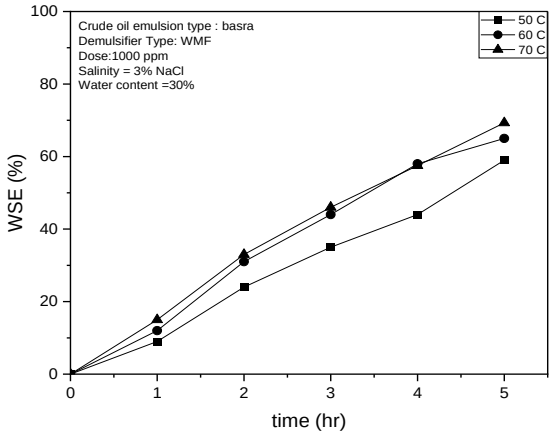

Figure 6. Temperature and emulsion separation efficiency.

3. 3. Assessment of Parameters Influencing the Demulsification Process

3. 3. 1. Impact of Settling Time and Demulsifier Doses on Water Separation Efficiency (WSE) Figure 5 depicts the average separation efficiency achieved at various concentrations of WMF (1000-6000 ppm) for the Basrah emulsion. The results indicate that water separation improves with longer settling times. This is because the presence of sufficient time increases the chance of oil droplets colliding with one another, increasing the phenomena of flocculation and coalescence of the oil into larger droplets. Demulsification and phase separation are both improved as a result (28). After 5 hours, the average separation efficiency reached 69.3% when the dose was 1000 ppm. Different responses were observed for other doses compared to the emulsion without the addition of WMF. So, the optimum separation time of the demulsification process was 5 h.

It was found, based on the outcomes, that increasing the concentration of demulsifier WMF led to a decrease in water separation efficiency (WSE), indicating an overdosing effect. Overdosing demulsifiers can lead to the restabilization of emulsions. Demulsifier lipophilicity or hydrophilicity and molecular mass determine the ideal demulsification quantity (15). This study found 1000 ppm optimal.

A decrease in demulsification efficiency can be traced to the production of a back emulsion, which is then labeled as a w/o/w emulsion because of the influence of shearing rate and temperature (25).

3. 3. 2. Effect of Temperature on Water Separation Efficiency (WSE) Experiments were run at varying temperatures of 50, 60, and 70 degrees Celsius, According to experts, demulsifiers are best studied at this temperature (20, 30). Oil, water, interfacial films, and the solubility of surfactants in both the oil and water phases are all impacted by temperature. Another key effect of temperature on emulsions is the decrease in viscosity that occurs as temperatures rise.

This is because the thermal energy of the droplets increases as the temperature rises (15, 31). For these reasons, investigating how temperature affects the stability of W/O emulsions is important. For varying concentrations of demulsifiers, rising temperatures were clearly associated with rising WSE. WMF performed best at 50, 60, and 70 degrees Celsius with a dosage of 1000 ppm and 5 hours of separation time, and the results are clarified in Figure 6 (57.2, 65, and 69.3%, respectively). As the temperature of the oil rises, its density and viscosity fall, allowing the water droplets to settle more quickly. So, the optimum temperature was 70 °C in this work.

3. 3. 3. Effect of Water Content The main factors affecting water-in-oil emulsion separation efficiency and

stability are water and oil content. A series of experiments were conducted to investigate the impact of water content and demulsifier concentration on water separation efficiency (WSE). The water content levels examined included 10%, 30%, and 50%, while the demulsifier employed was WMF. In Figure 7, it is observed that when WMF is present at 1000ppm, a temperature of 70℃, and a duration of 5 hours, Demulsification efficiency improves from 10% to 50% of the amount of water. Since the interface layer becomes thinner as the water content increases, the attraction of water droplets increases. The rate of coalescence consequently increases (7). However, due to the limited quantity of asphaltenes that can be adsorbed at the water-oil interface, the system tends to be inherently unstable. Specifically, As water content rises, the amount of asphaltene distributed per water molecule falls (23).

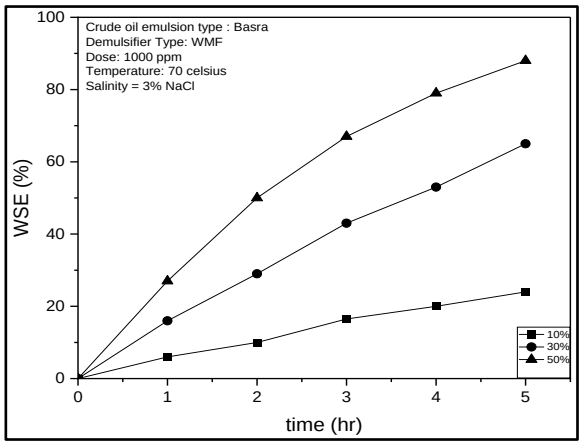

Figure 7. Water content and emulsion separation efficiency

4. CONCLUSIONS

The following conclusions may be drawn based on the study's findings:

1. The bio-demulsifier synthesized from waste corn oil shows promise as a potential and environmentally friendly alternative to chemical demulsifiers.
2. The synthesized bio-demulsifier has the potential to lessen the environmental impact associated with traditional demulsifiers. However, more research and development is needed to optimize the effectiveness of this novel bio-demulsifier, and achieve 100 percent water separation efficiency.
3. The characterization tests proved their efficiency in specifying and describing the bio-demulsifier.
4. Besides, these tests can be used to determine the types of functional groups they possess.

The maximum demulsification ratio was 69.3% at 1000 ppm, 5 hours of settling time, 70 °C, and 30% water content, all of which are operation factors with significant influence on the demulsification process.

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Persian Abstract

چکیده

علیرغم تنوع منابع انرژی در حال حاضر، آنها نتوانسته اند جایگزین واقعی نفت خام باشند، زیرا هنوز هم منبع اولیه انرژی در جهان محسوب می شود و تا سالیان دراز باقی خواهد ماند. همانطور که مشخص است، صنعت نفت شامل یک سری عملیات به هم پیوسته است که با استخراج نفت خام از چاه ها شروع می شود و به فرآیند پالایش آن ختم می شود. همانطور که مشخص است، صنعت نفت شامل یک سری عملیات به هم پیوسته است که با استخراج نفت خام از چاه ها شروع می شود و به فرآیند پالایش آن ختم می شود. این عملیات از نظر درجه سختی، هزینه و چالش هایی که با آن روبرو هستند متفاوت است. امولسیون نفت خام یکی از پرهزینه ترین مسائل در این صنعت مهم است. در مطالعه حاضر، یک بیودمولسیفایر دوستدار محیط زیست جدید سنتز شده از ضایعات روغن ذرت معرفی شده است. ویژگی‌های منحصربه‌فرد این دمولسیفایر زیستی جدید با استفاده از چندین آزمایش تشخیص داده شد، از جمله: طیف‌سنجی فروسرخ تبدیل فوریه(FTIR) ، کروماتوگرافی گازی–طیف‌سنجی جرمی (GC-MS)، تشدید مغناطیسی هسته‌ای پروتون ۱(HNMR)، و آنالیز وزنی گرما (TGA) فعالیت غیر امولسیون با استفاده از روش آزمایش بطری به عنوان تابعی از زمان ته نشینی، محتوای آب و دما ارزیابی شده است. حداکثر نسبت جداسازی ۶۹.۳٪ در دوز ۱۰۰۰ ppm به مدت ۵ ساعت و ۷۰ درجه سانتیگراد بود، در حالی که نسبت آب به روغن ۷۰/۳۰ بود. نتایج به‌دست‌آمده نشان می‌دهد که دمولسیفایر زیستی می‌تواند به‌عنوان یک جایگزین ایمن و سازگار با محیط‌زیست در واحدهای امولسیون‌زدایی اولیه مورد استفاده قرار گیرد که باعث کاهش مخاطرات زیست‌محیطی و هزینه‌های مالی مرتبط با صنعت نفت در هنگام استفاده از روش‌های دمولسی‌سازی سنتی می‌شود.