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**Continuous Separation Process of Water-in-Crude Oil Emulsion
by Simultaneous Application of an Electrical Field Combined
with a Novel Absorbent Based on Functionalised PolyHIPE
Polymer**

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Abstract

During the extraction process of crude oil, the removal of water from a high stability water-in-crude oil emulsions is critical in order to produce marketable product. Several technologies of separation exist today, e.g. stripping columns, centrifugal separators, coalescence separators, vacuum distillation systems and gravity separators, almost all of these approaches are not able to completely remove water from water-in-crude oil emulsions besides their high cost.

In this study, the preparation of a high internal phase emulsion (HIPE) was achieved on a laboratory scale. Subsequently, it was polymerized and sulphonated to produce a hydrophilic macroporous polyHIPE polymer (PHP) called silane (vinyl trimethoxy silane) PHP with a relatively high surface area of 104 m²/g. It demonstrates high water absorption capability in addition to its ability to remove surface active substances such as Mg, Ca, Na and Cl, from crude oil which cause crude oil emulsification.

The rates of demulsification of water-in-crude oil emulsions were examined in high AC field under various emulsion inlet flow rates from 100 ml/min to 1500 ml/min and different applied voltages from 1-5 kV (equivalent to 14-69 kV/m) by using a model of an electrostatic separator combined with silane PHP as absorber. It was found that the best separation efficiency was 91% with applied voltage of 5 kV and emulsion inlet flow rate of 100 ml/min. When the spent silane PHP was reused in the demulsification process under similar conditions, a separation efficiency of up to 73% was achieved. Also, it was noticed that the separation efficiency was increased

with the increase in applied voltage and reduction in the inlet flow rate of emulsion. Moreover, the original or spent silane PHP were able to remove the undesired metals present in the crude oil.

Keywords: Demulsification; Emulsion flow rate; Separation efficiency; Electrostatic Separator; Electric field strength.

عملية الفصل المستمر لمستحلب الماء في النفط الخام عن طريق التطبيق المتزامن لمجال كهربائي مع الماص بولي هايب بوليمر.

الخلاصة:

خلال عملية استخراج النفط الخام، تعتبر إزالة الماء من مستحلبات الماء في النفط الخام عالية الأستقرار أمراً بالغ الأهمية لإنتاج منتج قابل للتسويق. توجد العديد من تقنيات الفصل، على سبيل المثال أعمدة الفصل، فواصل الطرد المركزي، فواصل الأندماج، أنظمة التقطير الفراغي وفواصل الجاذبية، ولكن جميعها تقريبا غير قادرة على إزالة الماء بصورة كاملة من مستحلبات الماء في النفط الخام علاوة على تكلفتها المرتفعة.

في هذه الدراسة، تم تحضير مستحلب داخلي عالي الطور (هايب) مختبرياً. بعد ذلك، تمت بلمرته وسلفنته لإنتاج بوليمر بوليهايب عالي المسامية ومحب للماء وهو (فينيل تريميثوكسي سيلان) يسمى سيلان بوليهايب بمساحة سطحية عالية نسبياً تبلغ 104 م²/غم. يمتاز بقدرة عالية على امتصاص الماء بالإضافة إلى قدرته على إزالة المواد النشطة سطحياً من النفط الخام، والتي تسبب استحلاب النفط الخام مثل أيونات المغنسيوم و الكالسيوم والصوديوم والكلور.

تم تقييم معدلات الاستحلاب لمستحلب الماء في النفط الخام في حقل كهربائي متناوب عالي (أي سي) عند معدل تدفق متغير للمستحلب الداخل إلى الفاصل من 100 مل/دقيقة إلى 1500 مل/دقيقة وتطبيق فولتية متغيرة من 1-5 كيلو فولت (مايعادل 14-69 كيلو فولت) باستخدام فاصل الكترولستاتيكي بالتزامن مع استخدام سيلان بوليهايب كمتنص. وجد أن أفضل كفاءة فصل تم تحقيقها هي 91 % بتطبيق جهد كهربائي قدره 5 كيلو فولت ومعدل تدفق للمستحلب الداخل إلى الفاصل بمقدار 100 مل/دقيقة. عندما أعيد استخدام سيلان بوليهايب المستنفذ في عملية إزالة الاستحلاب تحت معايير مماثلة، تم تحقيق كفاءة فصل تصل إلى 73%. بمعنى آخر، لوحظ أن كفاءة الفصل عند استخدام السيلان بوليهايب الأصلي أو المستنفذ تزداد مع زيادة الجهد الكهربائي المطبق وتقليل معدل التدفق للمستحلب الداخل، بالإضافة إلى قدرتها على إزالة المعادن غير المرغوب فيها الموجودة في النفط الخام.

1. Introduction

The demulsification is an essential process which is mostly utilized for removing water and surface-active materials from crude oil emulsions in the oil industry. If the impurities like salts, water and metals ions comprised in the crude oil emulsion are not extracted, they cause numerous operational and commercial problems, which require attention and necessitates thorough studies to find suitable solutions. Amongst these problems, corrosion of pumps, pipes, tanks, and equipment are made

worse by the existence of contaminants like chloride. Moreover, the production of off-specification crude oil, failure of separation equipment in the units of the gas-oil separation, poisoning of catalysts, increase in the use of demulsifier(s), losses of production and increase in viscosity of crude oil in the transportation pipeline also necessitate further funds and cost of operations [1, 2]. The deposition of organics in processing equipment, inadvertently requires accommodating an increased volume from the presence of water to produce crude oil to conform with specifications. Therefore, due to these challenges, the crude oil emulsions are considered undesirable and the breaking of crude oil emulsions process of great importance and feasibility for crude oil producers. To be efficient, the content of water after demulsification process should be lower than 0.5% -3.0%.

Among the various demulsification methods, (e.g., sedimentation technique, thermal breaking methods, and centrifugation technique), the method of electrostatic demulsification is one of the simplest and more efficient demulsification methods [3, 4]. The process of electrostatic demulsification is basically based on the coalescence of water droplets and their precipitation. Although, the electrostatic approach has a comparatively large implementation, the electrical coalescence technique is known unwell due to electrical phenomena of interfacial polarization and hydrodynamic complexity. In general, the droplets coalescence includes three stages: (i) motion and approach of droplets because of the force of electrostatic; (ii) breakdown of the film detaching the droplets; (iii) collapse of the film and droplets coalescence. The effect of electrostatics is mainly connected to the first stage, that is, an increased the droplets motion rate in the electric field [5].

When applying an electric field, the droplets of water are polarized by the stimulated dipole. The fields of AC stimulate an oscillating motion of the droplets and the droplets of water convert into an ellipsoid, which leads to reduce the space between the droplets. It can be observed from the distribution lines of electric field that the intensity of the lines of field is increased between two adjacent droplets (Figure 1). The electrical potential gradient of the two droplets is proportional to the attractive force since the attractive force is proportional to the voltage gradient. Thus, the dense lines of the electrical field in Fig. 1 means the attractive force and gradient V

is very large. As the space between the water droplets decreases, they coalesce and turn into greater droplets. The collected droplets are possible to precipitate and the rate of precipitation of the droplets is proportionate to the diameter square of the droplet. In conjunction with what mentions above when applying an electric field, the following happens: (i) the particles of PHP in the water-in-crude oil emulsion contact a few numbers of water drop. This increases with time owing to Brownian motion of the water droplets and sedimentation; (ii) the polyHIPE hydrophilic surfaces permit the droplets to spread on the surface because of a small contact angle which breaks up the droplet and water absorbs into the PHP; (iii) the water is absorbed inside the particle and not freed back into the emulsion [6]. The high ability of water uptake and high surface area is crucial to remove as much water as possible.

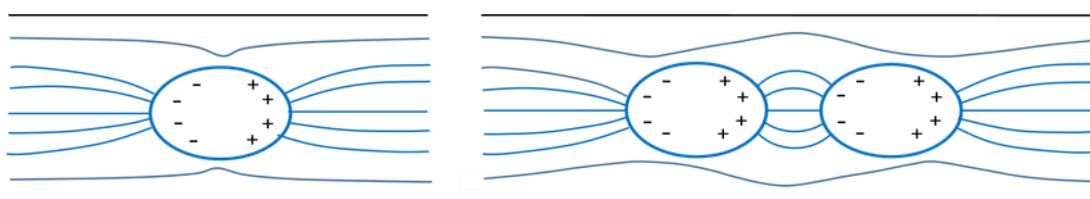


Fig. (1) Schematic diagram of induced dipole and interaction of dipole-dipole;
(a) Induced dipole. (b) Interaction of dipole-dipole.

Practically, both DC and AC fields can achieve an effective demulsification process. In DC fields, the electrophoretic droplet motion enhances the probability of coalescence, whilst in AC fields, the larger motion is essential to achieve greatest coalescence in bulk fluids [7]. Consequently, to attain an ideal continuous demulsification process, it is preferable to apply AC field, as it is more appropriate. The parameters which are considered to have an impact on kinetics of demulsification are (emulsion properties) interfacial tension, density, viscosity, surfactant concentration in oil phase, water droplet size, holdup, electrolyte concentration in water phase, frequency [8, 9]; (operating conditions) degree of mixing, shape, voltage, distance between the electrodes and temperature. In this research, a continuous electrostatic separator has been built, in which variable inlet flow rates of water-in-crude oil emulsion and AC voltages have been used to determine the demulsification rates.

This study aims to develop a continuous non-chemical demulsification method to enhance the efficiency of water separation process. Moreover, the removal of surface-active species from high stability water-in-crude oil emulsions depend on the use of a novel absorbent material is silane-PHP, combined with an electrostatic technique. It is an alternate method to the commonly used demulsification approach by adding chemicals, which leads to the formation of undesirable complex substances in addition to its high cost.

2. Demulsification of water-in-crude Oil Emulsion

All the past studies on demulsification under electrostatic force or demulsifier separately have been experimented in batch condition [4, 8]. While on continuous electrostatic demulsification a little research was reported [10, 11]. In this work, a continuous non-chemical demulsification method has been conducted by using a sulphonated silane-PHP as absorber combined with an electrostatic technique. This approach is like real-world process in order to increase the efficiency of the process of water separation and removal of the surface-active materials from water-in-crude oil emulsions of high stability. PolyHIPEs polymer, are a macroporous material with a highly porous interconnected monolithic structure. They consist of a well-defined, open-cell and uniform macrostructure with low dry density of around 0.1 gm/cm^3 [12, 13]. PHPs have numerous pretty features like a high ability for the disposal of metal ions through an ion exchange mechanism as well as the capacity to adsorb the toxins of organics due to the hydrophobic and hydrophilic domains on the porous structure walls [14, 15]. Moreover, the technique of electrostatic used has the suitable features to fulfill the aim of this research like low consumption of power owing to the low current usage during dispersion [16]. Furthermore, this technique does not risk the mechanical faults since it is independent of moving parts. Over and above, it has the capacity to be combined with various techniques such as, centrifugal separation, hydro cyclones, chemical emulsification, etc. to improve affirmative synergy for removal of water [17, 18]. This can be attained by creating the dispersed (water) phase droplets coalescence, which are stable by the indigenous surface-active materials existence in the emulsion such as oxides, phosphate, organic

carboxylates, asphaltenes and the like [19, 20], and gathering them in the silane-PHP absorber. The relative effect of the conditions on demulsification have been studied and found a significant increase in efficiency of separation because of the synergistic impact.

3. Experimental Work

3.1 Preparation of silane PHP

The procedure used to prepare the silane HIPE is reported by numerous researchers [21, 22] and is demonstrated in Figure 2. Silane HIPE contains two phases, namely, dispersed phase and continuous phase. The continuous phase (oil phase) consists of styrene monomer, divinylbenzene (DVB) crosslinker, sorbitan monooleate (Span 80) surfactant and vinyl trimethoxy silane (silane). Silane was used at various volume ratios of 10 %, 20 % and 30 % relative to styrene. Oil phase was prepared by adding these materials manually into the reactor with continuous mixing, where its volume is about 10 % (25 cm³) relative to the whole volume (250 cm³) of the silane HIPE emulsion. While the dispersed phase (aqueous phase) consisting of potassium persulphate as polymerisation initiator and double distilled water was mixed in a separate container, which presents around 90 % (225 cm³) of the total silane HIPE emulsion volume. Table 1 illustrate the volume percentage ratios for both continuous and dispersed phases.

The aqueous phase was pumped into the oil phase in the reactor with continuous mixing to produce silane HIPE. The operation time and mixing time were 10 minutes and 50 minutes, respectively. Then, the produced silane HIPE has been transported into 50 ml polypropylene containers with an inside diameter of 2.6 cm. These small containers were then placed in an oven of polymerization at 60 °C for 8 hours.

After polymerisation, the containers were removed from the oven, and then the silane PHP blocks were removed from the containers and cut into small discs by a sharp blade. After that, silane PHP discs were washed to eliminate the impurities from the pores and channels of the micro-porous structure and dried to remove the water of washing. Finally, the silane PHP surface was sulphonated using sulfuric

acid to convert it from hydrophobic to hydrophilic. Then, all the produced samples were evaluated in terms of surface area and ability to absorb water using Brunauer-Emmett-Teller (BET) method and water uptake test, respectively. It was found that the optimal PHP silane sample was 30% silane as it achieved a surface area of $104 \text{ m}^2/\text{g}$ and a high-water absorption capacity. Subsequently, they were processed and sieved to change them into granules with sizes ranging from 490 to $720 \mu\text{m}$ to use them as absorber in the separation experiments.



Fig. (2) Schematic diagram and picture of the mixing reactor used for HIPE preparation [21].

Table (1) Composition of the oil phase and the aqueous phase of silane HIPE.

	Compounds	vol., ml	vol. %	vol., ml	vol. %	vol., ml	vol. %
Oil phase	Styrene	16.50	66	14.00	56	11.50	46
	Divinylbenzene (DVB)	2.50	10	2.50	10	2.50	10
	Span 80	3.50	14	3.50	14	3.50	14
	Vinyl trimethoxy silane	2.50	10	5.00	20	7.5	30
Total		25	100	25	100	25	100
	Components	vol., ml		vol. %			
Aqueous phase	Distilled water (double)	222.75		99			
	Potassium presulphate	2.25		1			
Total		225		100			

3.2 Preparation the emulsion of water-in-crude oil

Different volumes of aqueous and crude oil phases were utilized to prepare water-in-crude emulsions. The aqueous phase was prepared by adding three kinds of salts (anhydrous powder forms), namely 0.6 g/l CaCl_2 , 5.0 g/l MgCl_2 and 28.1 g/l NaCl , to deionized water to mimic seawater. No artificial surfactants were added to stabilize the emulsions because of the natural surfactants and impurities that were already present in crude oil (i.e. resins, asphaltenes, wax, nitrogen, sulfur, oxygen, and some other elements mostly metals like vanadium, iron, nickel, copper etc.) and a density of 1.038 g/l was achieved.

A 40% (equivalent to 640 ml) aqueous phase was added into the 60 % (equivalent 960 ml) crude oil to prepare water-in-crude emulsions. Crude oil was placed in a container and mixed by a magnetic stirrer (Hiedolph instruments RZR 2052, Germany). Aqueous phase was added slowly into crude oil. Mixing was continued to compose the complete 40% water-in-crude oil emulsion (% by volume). The water-in-crude oil emulsion collected up to 1.6 L for continuous demulsification. Table 1 lists the several batches of the emulsions of water-in-crude oil that were prepared to get the high stability emulsion by utilizing different processing

parameters, namely mixing time and agitation speed. It was found that the emulsion-vii prepared with mixing speed of 2000 rpm and mixing time of 25 minutes was fully stable under the room temperature for about 122 days (by using a “bottle test”) which is a higher period time compared to the other prepared emulsions. Therefore, this emulsion was utilized in the separation experiments of the water/oil emulsions in this study. The range of the water droplets size dispersed in the crude oil has been investigated by a visual microscope and was 15-60 μm .

Table (2) Specifics of the prepared emulsions of water-in-crude oil.

Water-in-crude oil emulsion	Volume of aqueous phase (ml)	Volume of oil phase (ml)	Mixing speed (rpm)	Duration of mixing time (min)	Stability (day)
i	640	960	500	10	14
ii	640	960	500	20	21
iii	640	960	500	30	27
iv	640	960	500	40	36
v	640	960	500	60	37
vi	640	960	1000	60	71
vii	640	960	2000	25	122

3.3 Continuous electrostatic separator

0.5 gm of silane-PHP granules were added manually into 1000 ml of water-in-crude oil emulsion before sending the emulsion during the electrostatic separation vessel via a peristaltic pump and exposure to high voltage, followed by continuous mixing of the mixture by magnetic stirrer through the process of separation. Large pieces of silane-PHP which may be present in the emulsion are filtered by a steel mesh, while smaller pieces of silane-PHP pass through the separator and are assembled with the water phase. This is done to prevent clogging of the emulsion entrance.

The apparatus of demulsification, a continuous electrostatic separator, which is utilized in this research is illustrated in Figure (3). The electrostatic apparatus to carry out water-in-crude oil separation experiment was constructed using a perspex vessel with internal dimensions of 4.0 cm radius and 12 cm height. The parts of the vessel have been connected using a glue and plastic screws. The vessel was provided with upper and lower

copper electrodes. The electrodes have been utilized in the shape of copper plates with a thickness of 2 mm. The upper plate was not perforated and connected to the high voltage, while the bottom plate was perforated and connected to the earth to prevent short-circuiting. The bottom plate was fastened to the floor of the apparatus to obtain an electric field that incorporates most of the separator size compared to the cylindrical electrodes that give an electric field that does not incorporate most of the size of the apparatus of separation [23, 24]. Moreover, three circular center tubes (cavity) were installed on the apparatus with a diameter of 8 mm, which were used for the water-in-crude oil emulsion inlet, the demulsified emulsion outlet and water outlet, with three check valves utilized to control the water and emulsion flow rate. The separation cell includes a generator of AC high voltage (up to 5 kV) which was utilized to produce the fields of electric that created the coalescence of the droplets of dispersed water and their growth to larger droplets, which eventually drop freely due to gravity or electrostatic force and assembled at the apparatus bottom.

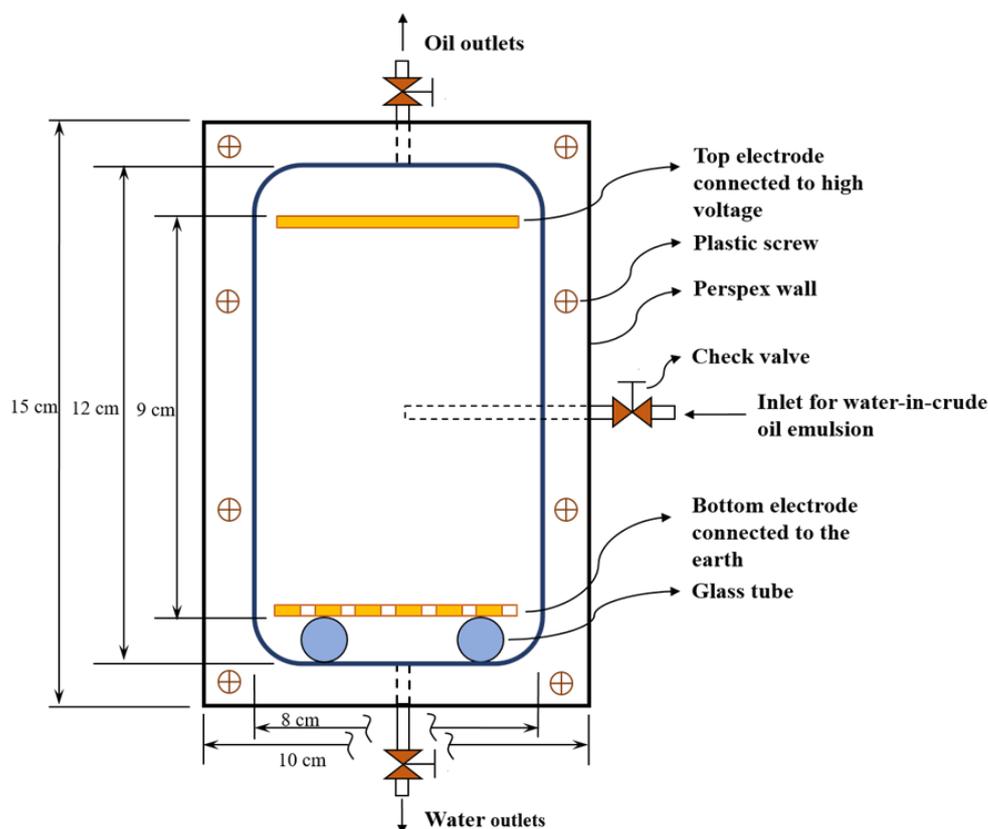


Fig. (3) Schematic diagram of the electrostatic separator.

4. Results and discussion

4.1 Measure the size of a water droplet and the range of pore sizes of silane PHP structure.

The size of water droplet of the crude oil emulsion was measured using an optical microscope called Olympus IX71 joined to a digital camera named an Olympus XM10. The microscope images were analyzed using ImageJ software. In each emulsion cut examined, a least of 200 droplets of water were measured and the mean size of droplet was calculated. The mean size of a water droplet was measured to be ~15 to 60 μm . The diameter of a stable W/O emulsion droplets for diesel oil was about 1 to 10 μm with a skewed normal distribution [25]. The average droplet volume of the water-in-crude oil emulsion is about 6 times greater than the average droplet volume of the W/O stable emulsion of diesel oil. This means that separating water from crude oil emulsions is easier than separating it from other hydrocarbon emulsions because its amalgamation and descent to the bottom of the separation vessel is easier due to the large size of water droplets.

To determine the pore size range of silane PHP, its microscope images were analyzed using ImageJ software. The pore sizes range was found to be 5.0 μm to 180 μm , which is consistent with the pore size range reported in previous studies (4.0 μm to 190 μm [21]).

4.2 Effect of inlet flow rate variation

The impact of the flow rate of emulsion on the efficiency of separation was assessed using the optimum sample of silane PHP produced. Experiments of separation were conducted over a high electric field strength ranging from 14 to 69 kV/m (1-5 kV applied along the distance of 0.09 m between the two separated electrodes). The inlet flow rate of the emulsion has been varied from 100 ml/min to 1500 ml/min. According to the performance of the process of continuous demulsification, the experiment results predicted that the separation efficiency of oil/water was significantly increased when the saline PHP absorber was added to the model of emulsion at a high-voltage electric field. In Figure (4), the variation of the degree of

separation is demonstrated as a function of inlet flow rate of the emulsion promptly after the passage of the emulsion over the electrostatic separator with absence and presence of silane PHP as absorber by utilizing a stable voltage of 5 kV (equal 69 kV/m) and different emulsion inlet flow rate. In the absence of silane PHP, when the rate of flow is over around 500 ml/min there is no prompt separation. Whilst the performance of separation with the existence of silane PHP was medium at a low inlet flow rate of the emulsion and low with a high inlet flow rate.

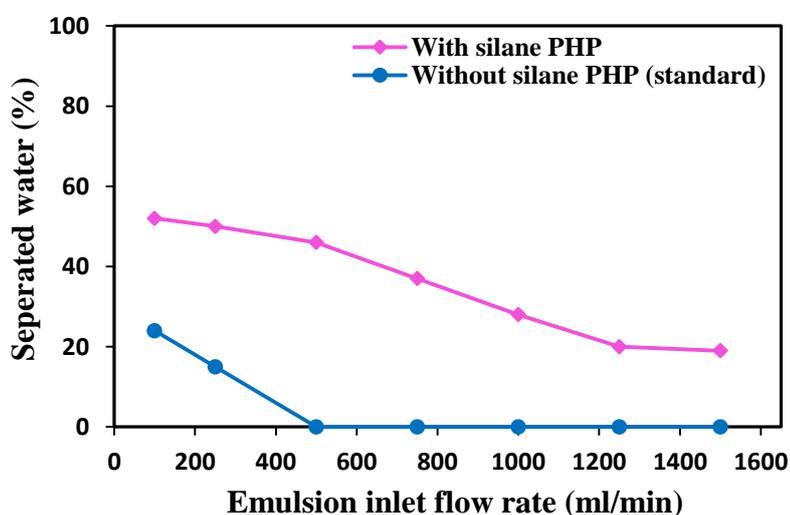


Fig. (4) Difference of percent water separated with emulsion flow rate through the electrostatic separator at 5.0 kV (69 kV.m⁻¹) applied voltage immediately after emerging from separator in the presence and absence of silane PHP.

Figure (5) and (6) demonstrate the separation efficiency after standing time of 1 and 24 hours, respectively with the presence of silane PHP absorber. The effect of silane PHP becomes unimportant as the standing time increases from 1 to 24 hours, whilst the impact is remarkable at relatively smallest flow rate as verified in these Figures. However, the outcomes that have been depicted show that the existence of silane PHP absorber and stable-strength electric field have promoted the process of demulsification, especially at a low flow rate of emulsion.

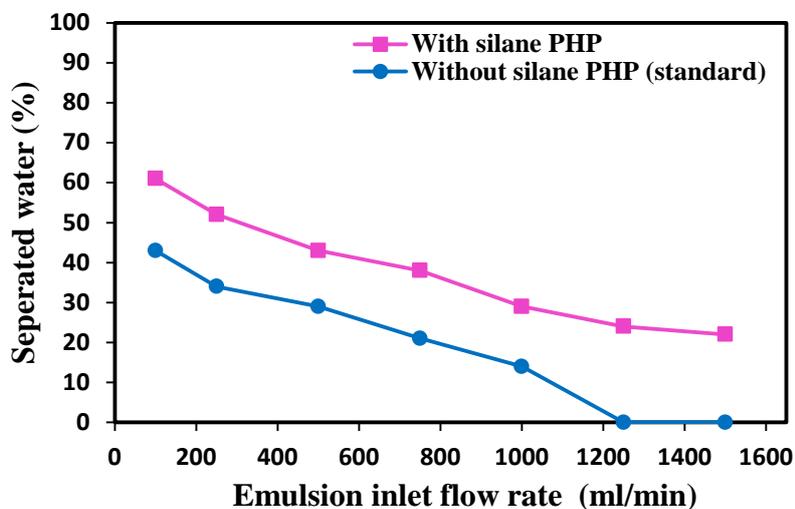


Fig. (5) Difference of percent water separated with emulsion flow rate through the electrostatic separator at 5.0 kV (69 kV.m-1) applied voltage, 1 hour after emerging from separator in the presence and absence of silane PHP.

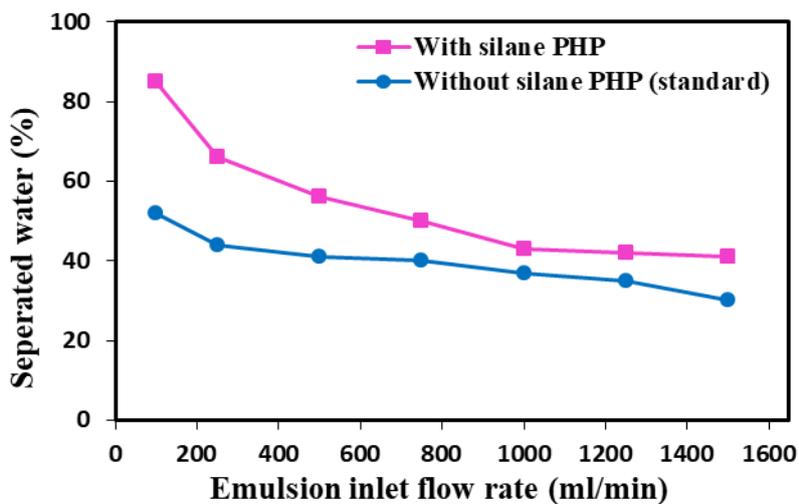


Fig. (6) Difference of percent water separated with emulsion flow rate through the electrostatic separator at 5.0 kV (69 kV.m-1) applied voltage, 24 hours after emerging from separator in the presence and absence of silane PHP.

4.3 Effect of electric field strength variation

The performance of silane PHP as an adsorber was investigated at various electric field strengths at a constant inlet flow rate of a 100 ml/min emulsion. Compared to the previous outcomes, a noticeable separation (more than 20%) occurs promptly after passage of the emulsion from the electrostatic separator with the presence of silane PHP as shown in Figure (7). However, the voltage value was at a minimal. While in the absence of silane PHP, no separation was observed under identical operating conditions. In fact, in the absence of silane PHP, the demulsification takes place via the electrostatic separator only when the strength of electric field is higher than 28 kV/m (equal to 2 kV).

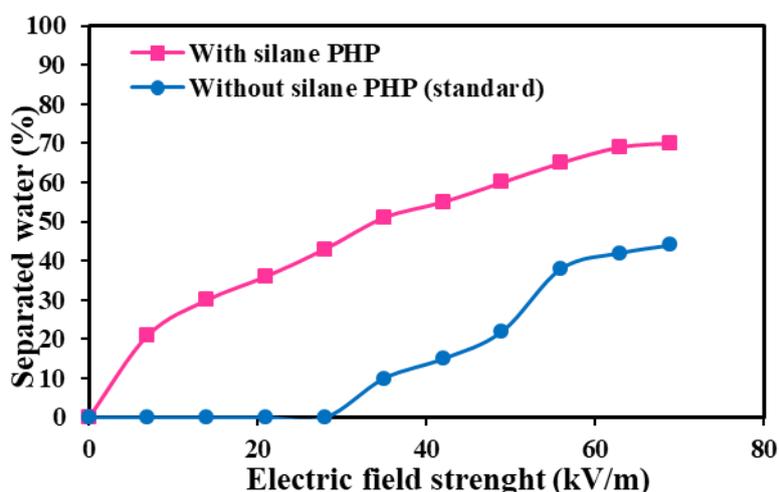


Fig. (7) Difference of percent water separated with electric field strength at constant flow rate of 100 ml/min, immediately after passing through the separator in the presence and absence of silane PHP.

After 1 hour of standing, a slight and moderate increase in separation efficiency was observed in the absence and presence of silane PHP emulsion respectively compared to the above immediate separation efficiency as shown in Figure (8). This means that the silane PHP has done a reasonable job in improving the performance of the demulsification process after an hour of standing.

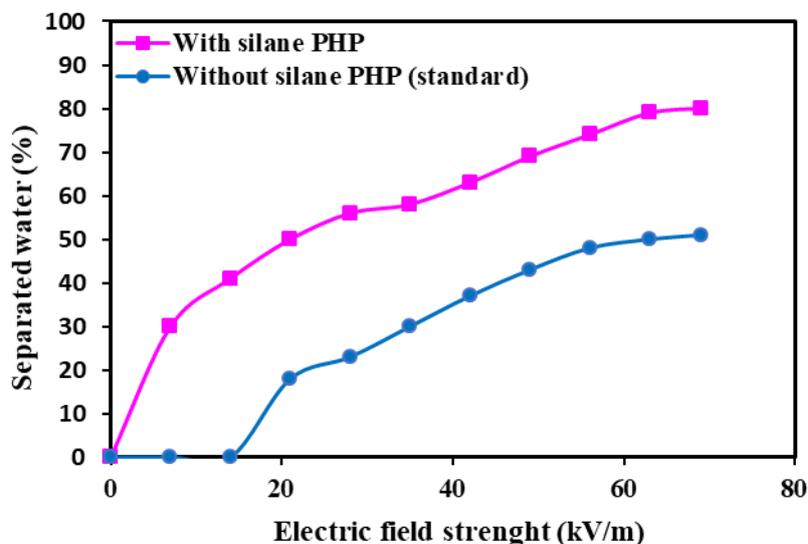


Fig. (8) Difference of percent water separated with electric field strength at constant flow rate of 100 ml/min, 1 hour after passing through the separator in the presence and absence of silane PHP.

It can be observed from Figure 9 that the demulsification process using a constant inlet flow rate of a 100 ml.min⁻¹ emulsion and electric field strength of a 5 kV (equivalent 69 kV/m) after 24 hours of standing that is achieved a separation efficiency of 56% in the absence of silane PHP. Interestingly, that the separation efficiency for the same condition in the presence of silane PHP achieved a high value, which reached about 91%. This percentage is considered very ambitious compared to the chemical separation method because both achieved almost the same separation percentage. Moreover, the advantage of this method could not produce polluting compounds as the case with the chemical separation method.

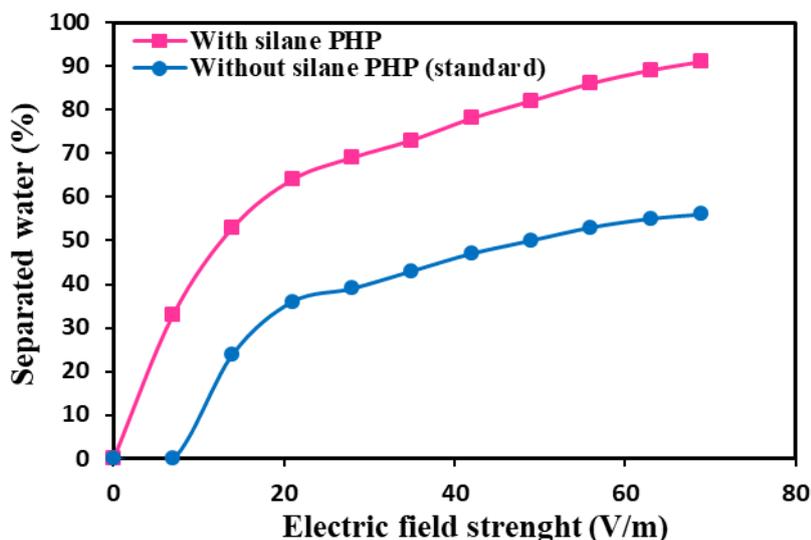


Fig. (1) Difference of percent water separated with electric field strength at constant flow rate of 100 ml/min, 24 hours after passing through the separator in the presence and absence of silane PHP.

4.4 Spent silane PHP reuse in experiments of separation using electrostatic field

In order to realize the efficiency of the spent silane PHP in water separation and the removal of active-surfaces from the water-in-crude oil emulsion, the separation experiments were carried out using the spent silane PHP combined with the electrostatic separator. The process of separation was conducted utilizing the water-in-crude oil emulsion at inlet flow rate of emulsion of 100 ml/min and changeable electric field strength (from 1 - 5 kV of applied voltage). It was noticed that the separation efficiency of original silane PHP, spent silane PHP and without silane PHP are 91, 73 and 56% of separated water, respectively, at the highest voltage as illustrated in Figure (10). The spent silane PHP has attained a higher efficiency of separation than the efficiency of separation in the absence of silane PHP, but it is less than the efficiency of separation of the original silane PHP.

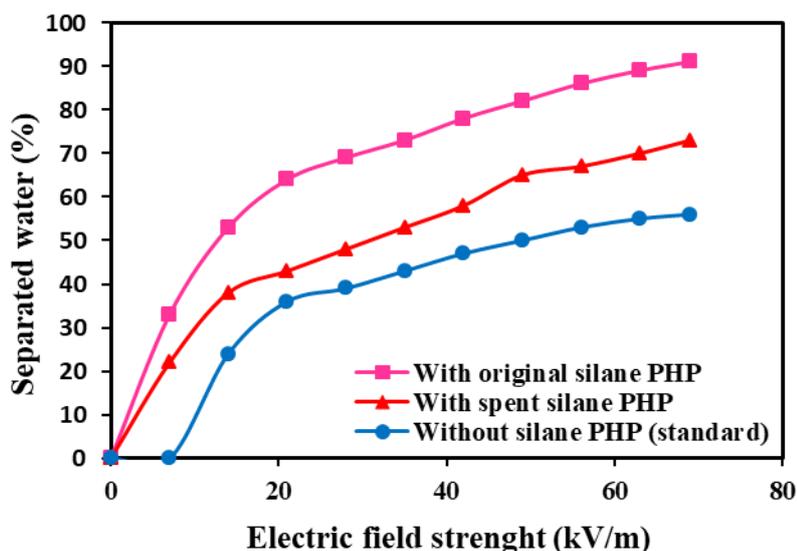


Fig. (10) EDX analysis result of reused spent silane PHP after demulsification, including the main elements adsorbed by silane PHP namely: Mg, Na, Cl and Ca.

In order to evaluate the elements in the spent silane PHP after the process of demulsification. The spent silane PHP was exposed to Energy Dispersive X-rays (EDX) spectroscopy and the results are summarized in Table (3). The EDX of silane PHP polymer denoted the existence of some more elements, such as Na, Mg, Ca and Cl, which were not present originally in the silane PHP but were joined onto silane PHP from the crude oil. This demonstrates that the silane PHP has capability to adsorb some other elements from the crude oil further to its capability to enhance the capacity of separation during the process of demulsification utilizing electrostatic field. Thus, silane PHP has the further feature attributed to its capacity to remove the metal ions like Mg, Ca, Na and Cl that induce crude oil emulsification by an ion exchange mechanism.

Table (3) EDX analysis result of silane PHP after demulsification, including the main elements adsorbed by silane PHP namely: Mg, Na, Cl and Ca.

Element	Weight %	Atomic %
C	83.45	89.32
O	8.71	6.94
Si	0.64	0.32
S	3.94	1.62
Cl	0.91	0.41
Ca	0.16	0.07
Na	1.97	1.15
Mg	0.22	0.17
Total	100.00	100.00

With the aim to verify the elements in the spent silane PHP structure after the process of demulsification, the spent silane PHP has been subjected to EDX spectroscopy. The outcome is presented in Table (4). It can be observed that the spent silane PHP included elements which were not present in the original PHP-I, namely Na, Mg and Ca, that were adsorbed from the crude oil. This shows that the spent silane PHP also has sufficient capacity to remove the undesired elements from the crude oil which cause the crude oil emulsification. Thus, the spent silane PHP also has the ability to remove the existing undesired metals in the crude oil but with lower efficiency than in the original silane PHP.

Table (4) EDX analysis result of reused spent silane PHP after demulsification, including the main elements adsorbed by silane PHP namely: Mg, Na, Cl and Ca.

Element	Weight %	Atomic %
C	84.01	89.74
O	8.56	6.83
Si	0.65	0.29
S	3.95	1.65
Cl	0.77	0.28
Ca	0.14	0.06
Na	1.71	1.03
Mg	0.21	0.12
Total	100.00	100.00

Silane PHP has been analyzed by Environmental Scanning Electron Microscope (SEM). Figure (11, a) displays the SEM micrograph of silane PHP before using it in the separation experiment. While, in Figure (11, b), the image of SEM clarifies the

appearance of spent silane PHP which has been utilized in separation experiments. After finishing the process of demulsification, silane PHP was gathered, dried, and sent to the SEM for analysis to evaluate its capability to remove the surface-active materials. It can be observed that the structure of porous silane PHP partially collapses as an effect of a mechanical stresses produced from pumping of emulsion along with the process of drying and shows a few clogged pores due to the accumulation of surface-active material absorbed by the silane PHP inside the pores.

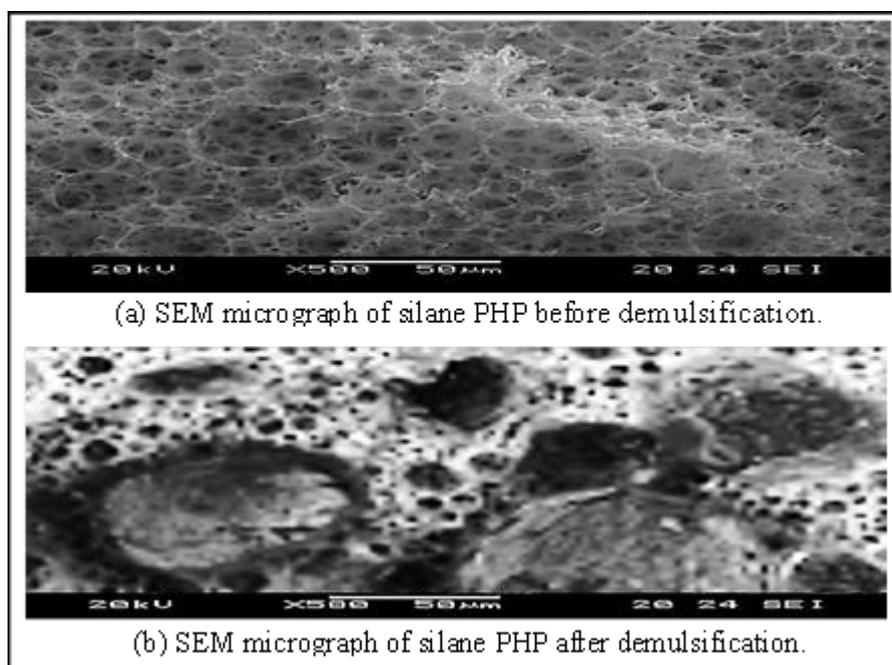


Fig. (11) SEM micrograph of the silane PHP (a) before the separation experiment (at 500x magnification) and (b) after the separation experiment (at 500x magnification).

5. Conclusion

The process of demulsification in recovery of crude oil was simulated with the electric field strength, the inlet flow rate of the emulsion and silane PHP absorber. A model of water-in-crude oil emulsion has been prepared with 40% of water and addition of certain concentrations of calcium, sodium, and magnesium salts to make it more stable. The size of the water droplets was around 15-60 μm and was stable for around 122 days at room temperature.

Using a continuous electrostatic separator combined with original and spent silane PHP, the separation efficiencies of demulsification process of water-in-crude oil emulsion with different inlet flow rate and high AC electric fields were investigated.

The efficiency of water separation increases with the increase in the applied electric field due to generation of an electrostatic force between water droplets which distort along the field direction. The addition of silane PHP absorber to water-in-crude oil emulsion increases the water separation as it helps to accelerate the film collapse around the water droplets as well as in its adsorption of water inside the pores.

The findings from a series of demulsification experiments under various operating conditions reveal that the separation efficiency using the inlet flow rate of 100 ml.min⁻¹ and applied electric field strength of 5 kV with the presence of the original and spent silane PHP were 91% and 73%, respectively. These separation efficiencies are higher than as compared to the separation efficiency in the absence of silane PHP of 56%. This means the original silane PHP achieved a significant separation efficiency, while spent silane PHP attained a satisfactory separation efficiency. Moreover, they achieved a high capacity to remove surface-active materials, such as Mg, Na, Cl and Ca ions, from crude oil which cause the emulsification of crude oil. Finally, it was found that the silane PHP absorber attained sensible results in terms of the water separation efficiency and the ability to remove surface-active species from water-in-crude oil emulsion, but needs some chemical modification to attain more reasonable results for multi-used.

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