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SUSTAINABLE LIMONENE-BASED ORGANIC DEMULSIFIER

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ABSTRACT
Crude oils are the most important products in the world as that would later be used to produce petroleum. For many years, the petroleum industry has always faced a challenge in oil production which is oil production being accompanied by water. The water that is being discovered in the oil production could be discovered in two forms which would either be the free water or in the form of emulsion. This research is being conducted to investigate and propose a solution on demulsifying water from oil production. Limonene is being used for the demulsifying process. The limonene is being obtained from citrus fruit peels as an eco- friendly demulsify. Additional agents are being added to the limonene to improve effectiveness. The outcome of this investigation shows the organic demulsifies have effectively separated the emulsions. This research has emphasized limonene based organic demulsifies as a solution on separating water in oil emulsion. This would allow the crude oil production to provide both operational efficiency and environmental sustainability.
Keywords: crude oil, petroleum, emulsion, demulsifies, oil production, Limonene, free water.

Introduction

Crude oil is one of the most precious commodities in the world that petroleum engineers make it a duty to design and develop methods for extraction. The quality level of oil that is produced is a selling point for the oil industry. This makes it a necessity that oil produced is in its best state as much as possible. For many years of oil production, one of the major challenges that petroleum engineers encounter is oil production accompanied with water. It is very common to have oil production accompanied with water production from the formation. The water that is produced alongside oil can exist in two forms. The water can exist as free water or be produced in the form of emulsion. Having produced water exists as free water means that the water is produced and settles out quickly or rapidly. This water produced can be dealt with more easily because it makes a distinctive separation. Water that is produced in the form of emulsion however is a more complicated problem.

One of the major challenges in the oil and gas industry is the issues (W/O Emulsion issue) arising during the production and transportation of crude oil. Emulsification can happen inside the reservoir when crude oil and water is squeezed through the narrow pores in the reservoir or because of vigorous mixing of oil and water during oil production. Emulsions occur in almost every stage of oil production. The stability of emulsions however is very field dependent and there would be varying characteristics for different fields. Emulsions can be formed when there is contact between the oil and water either in the reservoir, by turbulence during the mixing of the immiscible fluids when being produced or when there is the presence of an emulsifying compound in the crude oil, for example, asphaltenes.

Organic demulsifier is one of the best solutions to prevent environmental pollution without diminishing its main functions of emulsion breaker. An organic demulsifier is a great approach to reducing the negative impact chemical demulsifiers have on the environment without diminishing its main function of breaking the emulsions formed. The use of local raw materials to create organic demulsifiers would have a lesser negative impact on the environment. Considering environmental constraints from chemical demulsifiers, it would be great to have a more environmentally friendly, biodegradable demulsifier. Limonene is an oil extract from citrus fruits peels which has tendencies of breaking emulsions. This research entails the investigation of limonene as an emulsion breaker and an organic demulsifier.

Problem Identification on Sustainability of Production

Emulsions cause the malfunctioning of pumps reduces oil quality and generally decreases oil production. Lower levels of quality of the oils affect the prices of the oil resulting to economic loss and more challenges in the operational sector. As mentioned earlier, chemical demulsification is the most widely used demulsification technique in the petroleum industry. The over dependence on chemical demulsifiers has brought to the attention how the chemical demulsifiers are to an extent toxic to nature. Most of the conventional chemical demulsifiers are effective in resolving water-in-oil emulsions but their application is restricted due to environmental concerns. The chemical demulsifiers are toxic and may cause serious environmental degradation during water disposal (Abu et al.,2016). They cause water pollution and human health hazards. Chemicals in chemical demulsifiers are not eco-friendly, toxic, and expensive. Regardless of effectiveness of chemical demulsifiers in destabilizing emulsions, the drawbacks it has can be improved with an organic demulsifier.

In addition to crude oil, a secondary common component of produced water is synthetic chemicals, which are often referred to as production chemicals because they are added to assist in the oil production process. They serve a range of purposes and have a correspondingly wide range of chemical compositions, although their precise formulations are often undisclosed, even to the producers that use them, as they are proprietary to the chemical manufacturers. The concentration of most production chemicals is low in treated produced water. Discharge of treated water is continuous during production and the slow accumulation of these nondegradable chemicals in the marine life after each disposal eventually becomes extremely toxic. The aim of this research project was to formulate an environmentally friendly limonene-based organic demulsifier to break water in oil emulsion based on the following objectives:

1. To propose an organic demulsifier to break water in oil emulsion.
2. To devise the methodology for the development of the organic demulsifier.
3. To compare the organic and synthetic demulsifier in terms of effectiveness.

Overall System Block Diagram

The procedures involved in the overall block diagram are similar to the procedures other researchers had performed to create an organic demulsifier. The materials that were used in formulating the organic demulsified. Figure 1 shows the overall block diagram of the system.

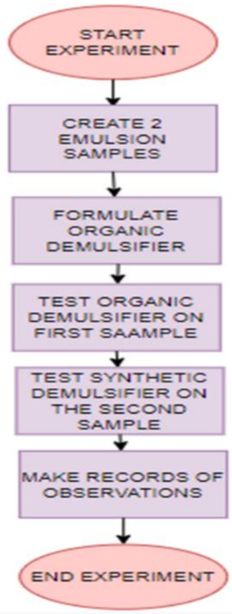

Fig 1: Overall block diagram

Creation of Synthetic Emulsion

In the block diagram, the experiment started by creating synthetic emulsion so that the demulsifiers can be tested. The demulsifier works when it breaks emulsion. It had been mentioned earlier that to prepare the emulsion the oil and water ratio was kept at a 70:30 percentage ratio. The greater percentage was oil. The oil that was used was heavy waxy crude oil. A very high viscous oil that could not flow under normal room conditions. The oil was semi-solid at room temperature and was melted into liquid in order to mix with water to create the emulsions. The heating and melting process of the waxy crude oil took a total of 4 hours in an oven kept at a temperature of 176°F. This temperature was appropriate for melting the wax without boiling it or causing it to evaporate. The waxy crude oil was kept in a glass bottle inside the oven. This glass bottle after every hour, was taken out of the oven, gently shaken, and placed back in the oven. This was to ensure that there was a heat equilibrium within the glass bottle to allow the wax melt properly throughout the bottle. The oil was ready to be used after melting properly.

All emulsions prepared in the laboratory were kept in a bottle sample for settling and observation purposes. A bottle sample can hold up to 50ml of volume. Following the 70:30 percentage ratio, melted oil of 35ml was mixed with 15ml of distilled water to create the emulsion. There are many ways of producing an emulsion and it is usually achieved by applying mechanical energy through agitation, normally by using a homogenizer. Initially, the interface between the two phases is deformed and large droplets are formed. These droplets are subsequently broken up into smaller droplets by the continuing agitation. (Pieter,1993). To create the emulsion, the oil and water were kept in a homogenizer at a rotation of 1200 rpm.

The speed was kept high because a greater agitation would result in a more stable formation of emulsion. The whole process was kept in a hot water bath. This was to maintain the heat in the oil to prevent the oil from hardening back into wax. The emulsion creation process took a total of 15 minutes. By the last minute of the 15minutes, the setup was taken out of the hot water bath and the oil-water-mixture was transferred into the bottle sample and allowed to settle. Allowing sample settling enhances stabilization of emulsions. The oil-water-mixture could settle for a total of 24 hours with each hour, observing the bottle sample to see if there were any changes. In the first 3 hours of observations, the sample had settled and there was a distinct separation in layers of the fluid. The layers were oil, emulsion, and water at the bottom of the bottle. There were no differences in observation made after the 4th hour and that remained same until the 24th hour of observation. The oil in the mixture had also turned back to wax.

The total volume recorded after the oil water mixture was transferred into the bottle sample was 35ml for both bottle samples. After the samples were left to settle the volumes each layer occupied were marked and recorded as the bottle sample was calibrated. For identification purposes, first oil water mixture that was transferred into the bottle sample was named Bottle Test A and the second sample, Bottle test B. The changes in volumes with time are recorded and noted in Table 1 and Table 2.

Table 1: Bottle Test A layer recordings

LAYERS	LAYER VOLUMES (ml)				
	AFTER 1 HOUR	AFTER 2 HOURS	AFTER 3 HOURS	AFTER 4 HOURS	AFTER 24 HOURS
OIL	15	14	13	13	13
WATER-IN-OIL EMULSION	15	17	18	20.8	20.8
WATER	5	4	4	1.2	1.2

Table 2: Bottle Test B layer recordings

LAYERS	LAYER VOLUMES (ml)				
	AFTER 1 HOUR	AFTER 2 HOURS	AFTER 3 HOURS	AFTER 4 HOURS	AFTER 24 HOURS
OIL	19	21	22	22.5	22.5
WATER-IN-OIL EMULSION	11	9	8	7.5	7.5
WATER	5	5	5	5	5

Formulation of Organic Demulsifier

The demulsifier being formulated is a limonene based organic demulsifier. B1 to B5 are blending agents that are mixed with the lemon extract to enhance the efficiency of the organic demulsifier. In each sample of organic demulsifier formulated, EB is the outcome after E1 has been mixed well with B1-B5. This is to say that, for a sample of organic demulsifier to be formulated, measured portions of E1, B1, B2, B3, B4 and B5 are used and the final outcome of the mixture is EB.

To begin the formulation, E1 was extracted. Liquid juice from lemon was the best source of limonene for the purpose of the project. The extract was acquired by obtaining the juice from fresh lemons. The juice is kept fresh and kept in a beaker, B1 to B5 are all added to create the mixture. The measurements of each material that was used are in Table 3.

Table 3: Material Measurements

Labels	Material	Amount
E1	Limonene	15ml
B1	Palm oil	8ml
B2	Calcium Hydroxide	3g
B3	Olive Castile soap	40ml
B4	Tapioca starch	20g
B5	Distilled Water	20ml

The tapioca starch that was added was to enhance the water resistance of the organic demulsifier. The demulsifier must have an ability to resist water in the emulsion so that water does not break the demulsifier even before it starts working. Water was added as a solvent and distilled water was most preferable to reduce the chances of having impurities in the demulsifier. Palm oil was added to enhance the emulsion breaking ability of the demulsifier. Olive castile soap was also added to enhance the emulsion breaking ability of the demulsifier since it acts as a surfactant. Calcium hydroxide was added to bind the blending materials together so that they do not form clamps in the mixture. The blending agents were finally added to the extract and the organic demulsifier was transferred into a bottle sample. The blending agents must be in a homogeneous blend to be added to the lemon extract. If the blending agents were unable to be in a homogeneous mixture, they would have been changed as shown that is the alternative step to follow in such a condition.

Testing Demulsifiers

The next step after organic demulsifier formulation was to test the demulsifiers on the emulsions that were created. The oil in the bottle samples had completely solidified however the water-in-oil emulsion layers were very visible. In order to test the demulsifier on the emulsions, the bottle samples had to be heated up to get the waxy oil back in a liquid state so that the demulsifier could sink through to reach the emulsions. Since the first layer in the bottle sample was waxy crude oil, there was no way the demulsifiers could penetrate through to reach the emulsion and break it. The bottle samples, in their racks, were directly placed into a preheated oven at a temperature of 176°F. The bottle samples were kept in the racks in an upright position to prevent the layers from mistakenly mixing up.

The bottle samples were kept in the oven for an hour. Every 15 minutes of that hour was used to check on the bottle samples. At the end of the hour, the bottle samples still in the racks were taken out and quickly transferred into a hot water bath. The rack was kept in the hot water bath to maintain the heat in the bottle samples to prevent the oil in the bottle samples from hardening up back into wax. The samples were ready to be tested on with the demulsifier.

Testing Organic Demulsifier

The materials in the organic demulsifier had already settled and distinct layers in the bottle sample could be seen. The bottle was shaken for 2 minutes to ensure that all the materials mixed up properly to have one consistency before it was tested on the emulsion. With the emulsion bottle samples still in the hot water bath, the organic demulsifier was added to the emulsion bottle sample. Bottle Test A was assigned to the organic demulsifier. The demulsifier was added in small amounts of 0.2ml with the help of the dropper into Bottle Test A and as it slowly moved to the emulsion layer, keen observations were made. 0.2ml of the organic demulsifier being added to the emulsion bottle sample had already caused the emulsion to slowly disintegrate. An extra volume of 0.2ml was added and there was more disintegration of the emulsion. Still under keen observation, 0.2ml more of the demulsifier was added to the sample and the rate of disintegration had increased at this point. The emulsion was slowly breaking apart as the demulsifier travelled through it.

The oil and water in the emulsion layer were being separated. An extra volume of 0.2ml of the organic demulsifier was once again dropped into the sample and there was no significant change in the disintegration rate of the emulsion. The water-in-oil emulsion was being disintegrated, however, additional volume of the organic demulsifier was not making any difference in how fast it was being disintegrated. After 180 seconds of observation, the emulsions had been completely broken. There had been successful separation of the water-in-oil mixture. A total volume of 0.8ml was used to separate the water-in-oil emulsion that was present in Bottle Test.

Constructional Flow

The work of the organic demulsifier that has been formulated is to break emulsions that are created because water and oil cannot mix up. An emulsion has to be present for one to be able to tell if a demulsifier is effective. When the emulsion was created and the demulsifier was formulated, the demulsifier could be tested. The demulsifier would work in breaking the emulsions which would cause the separation of oil and water back into 2 different fluids. Figure 2 shows how the entire system is connected and how each step is related to each other in the entire system implementation.

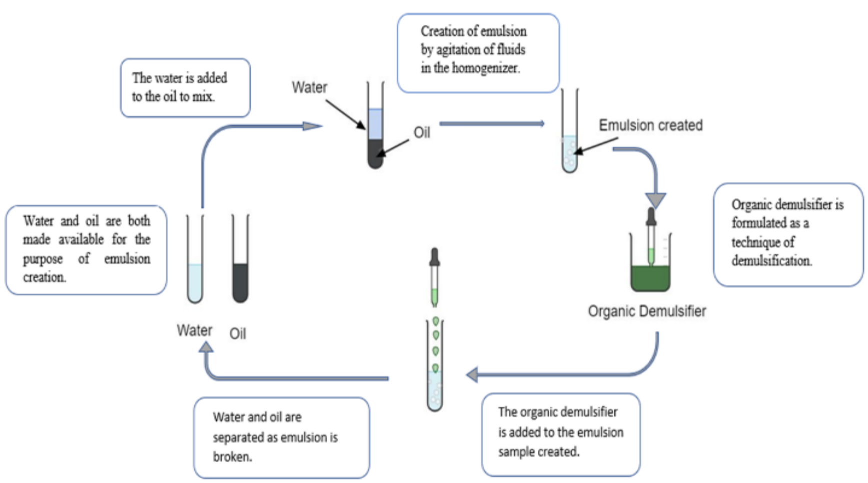

Fig 2: Connection of entire system

Working Principle

The working principle of the entire system is explained with the help of a flowchart that is shown in Figure 3.

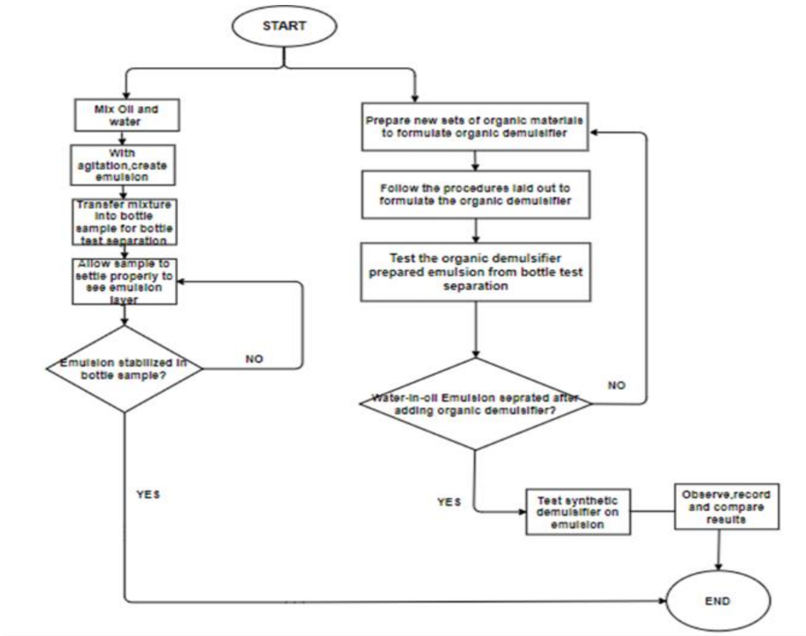

Fig 3: Working principle

Bottle Test Emulsion Separation

Figure 4 shows the bottle test separation of the oil water mixture after it had been allowed to settle. It can be seen that the total volume of the oil water mixture amounts to a total of 35ml. The darkest layer at the uppermost top is the oil. Beneath the oil layer is the muddy looking brown layer which is the emulsion that has been created. Oil and water have been trapped in this layer. This is the layer of concern as the demulsifier that has been formulated separates the oil and the water from the mixture that is trapped in that layer. Beneath the muddy looking layer of emulsion is the slightly clear layer of water at the bottom of the bottle sample.

Fig 4: Bottle Test A before Demulsification

Emulsion Separation with Organic Demulsifier

Bottle Test was used to test the effectiveness of the organic demulsifier that had been formulated. The emulsion that was present had trapped an amount of water and oil. Addition of the organic demulsifier into the bottle was able to disintegrate the emulsion. Figure 5 shows that when the organic demulsifier was added to the sample, the layer of emulsion that was between the water and the oil had been fully separated. There is no longer a layer of emulsion. Only 2 layers of fluids can be seen.

At the top is the oil and at the bottom is the separated water mixed with the organic demulsifier that was added. The organic demulsifier was able to successfully separate the oil and water from the mixture and fully break the emulsion layer that was between the two fluids. The oil that was trapped in the emulsion layer has returned into the oil layer and the water that was trapped in the emulsion layer has also returned to the bottom of the bottle. It had been mentioned earlier that the bottle sample containing the emulsion was heated before the demulsifier was tested on it. Before the organic demulsifier was added to the bottle sample, the volume of oil, not regarding the ones trapped in the emulsion, was marked at a volume of 13ml but after the demulsification process, the volume of oil was marked at a volume of 25ml. The demulsification process had released a volume of 12ml of oil that was trapped in the emulsion. At the bottom of the bottle sample is a mixture of water and demulsifier. The mixture is reading at a volume of 13 ml indicating that volume of water that was in the oil water mixture before sample settling was 11ml. Table 4 simplifies the final volume recorded after demulsification with the organic demulsifier.

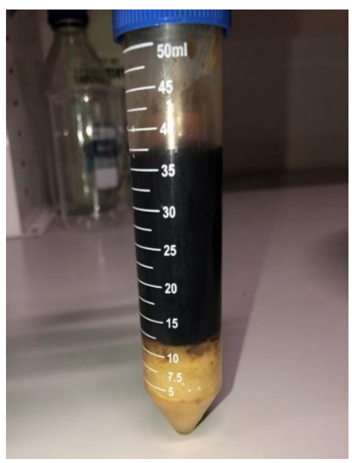

Fig 5: Bottle Test A after Demulsification

Table 4: Water and Oil Layer Readings

Layer	Oil(ml)	Water (ml)
Before demulsification	13	1.2
After demulsification	25	12.2

Increase in volume of both oil and water is as a result of separation of the emulsion. The emulsion had trapped this much volume of both oil and water. This is how emulsion plays a role in reducing the total volume of oil that is acquired from the reservoir.

The results that were obtained from the demulsification process would be compared based on

I. Rate of emulsion separation.
II. Amount of demulsifier used.
III. Oil recovery from emulsion.
IV. Water removal efficiency.

Rate of Emulsion Separation

The chemical demulsifier that was used separated the emulsions faster than that of the organic demulsifier. After each drop of the chemical demulsifier into the bottle sample, an immediate disintegration of emulsion was observed. However, the organic demulsifier slowly separated the emulsion as the drops were being added. The demulsifier was seen to travel slowly between the emulsion. The organic demulsifier separated the emulsion in 180 seconds while the chemical demulsifier separated the emulsion in 68 seconds.

Amount of Demulsifier Used.

1.2ml was the volume of chemical demulsifier that was used to separate the emulsion while the volume of organic demulsifier used was 0.8ml. There was more chemical demulsifier needed to break emulsions but the organic demulsifier did the separation just fine with a lesser amount.

Oil recovery from emulsion.

The chemical demulsification did not yield in any oil recovery after the emulsion was separated. It worked as if it took the emulsion away with the oil trapped in it. The organic demulsifier however, showed a very good amount of oil recovery that was trapped in the emulsion after separation. From oil volume of 13ml, a total of 25ml of oil was obtained after organic demulsification resulting in 12ml of oil recovery which is about 52% recovery of total oil in the bottle sample. The volume of oil increased after organic demulsifier was used and no oil recovered after chemical demulsification

Table 6: Comparison between chemical and organic demulsification

RESULTS	CHEMICAL DEMULSIFICATION	ORGANIC DEMULSIFICATION
Ability to separate emulsion	Separates emulsions completely	Separates emulsions completely
Volume of demulsifier used	1.2ml was used in the demulsification process	0.8ml was used in the demulsification process
Oil recovery from emulsion	There was no oil recovery from emulsion after demulsification	There was an oil recovery of 52% from the emulsion after demulsification
Water Separation efficiency(wt.100%)	The water separation efficiency of chemical demulsifier is 67%	The water separation efficiency of organic chemical demulsifier is 90%

Testing of the proposed design.

Formulation of the organic demulsifier was completed within 8 minutes. The organic materials that were needed to formulate the organic demulsifier had been made ready, so it was a matter of measuring, mixing, and transferring into the bottle sample to be used. The demulsifier formulated came out with a yellowish colour and that is prior to the usage of palm oil. The formulation had a nice but strong smell. Limonene is heavily scented and because of that the demulsifier was an orange scented formulation. The demulsifier was not highly viscous. The flow of evaporated milk the demulsifier could be compared to that of the demulsifier. Figure 6 shows the final outcome of the organic demulsifier after it had been formulated and transferred into the bottle sample. The demulsifier was formulated and tested on the emulsions. The results recorded are explained in the previous chapter.

Fig 6: Formulated Organic Demulsifier

The data provided is plotted in Figure 7.

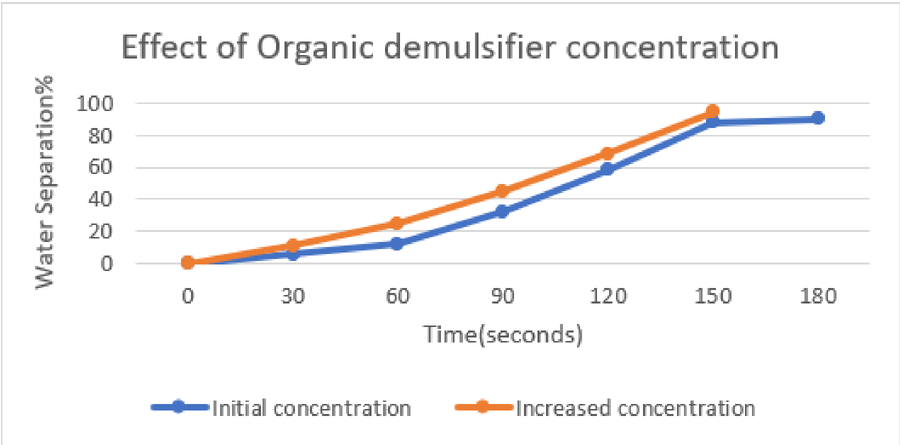

Fig 7: Effect of organic demulsifier concentration on emulsion separation

Biodegradability Testing.

Biodegradability can be referred to as the ability of a material to break down or decompose to reduce pollution. This is the ability of that material to disintegrate by bacteria or microorganisms acting on it. The decomposition process is usually within a year. There is usually no ecological harm during a biodegradability process. The demulsifier biodegradability testing is a test that is performed to help evaluate the biodegradation potential of the organic demulsifier formulated. Emulsion separation separates water and oil. When the water is separated from the emulsion, the organic demulsifier is remained inside the water separated. In an oil treatment operation, water that is separated from emulsions are treated and returned back to water sources. For this test, organic demulsifier is added to emulsion sample to break the emulsion. When the emulsion is separated, the water at the bottom of the bottle sample contains the water that is separated and the organic demulsifier that is added. That is the portion of interest for this test. 1ml of organic demulsifier is used for this test and is expected to reduce with time as biodegradation begin. The setup is checked every 30 days to record changes. The results recorded during observation is tabulated in Table 10.

Table 10: Volume of demulsifier during biodegradation

DAY	Volume of Demulsifier(ml)
1	1
30	1
60	1
90	0.95
120	0.9

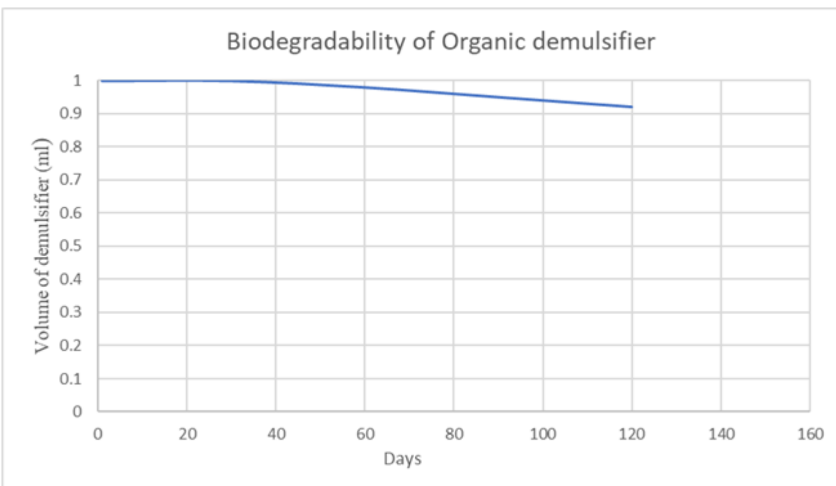

Fig 8: Biodegradability of organic demulsifier

The demulsifier started to reduce in volume as the time increased. This shows that the demulsifier is able to degrade on its own with given time. At 120 days, the demulsifier in the water had reduced.

Sustainable Development and Environmental considerations

Beginning with environmental sustainability, the materials that are used to formulate the organic demulsifier are organic and local. They are organic materials that can rot and return back to living matter. In chapter 5, in test 3,the biodegradability of the organic demulsifier was tested. It was observed that with time, the organic demulsifier started biodegrading and reducing. This characteristic of the organic demulsifier makes the organic demulsifier achieve environmental sustainability. The organic demulsifier has the ability to get rid of itself with time. This means that with a given time, organic demulsifier would not contribute to waste in the environment. The continuous break down of demulsifier would prevent it from piling up in the environment.

The formulated organic demulsifier contributes to waste minimization in the environment. This contributes to environmental sustainability. The organic demulsifier does not cause pollution air pollution either. In Test 4,it was seen that concentration of organic demulsifier changed the pH of water from 5 to 13. The organic demulsifier does not make water acidic which is a good characteristic. Even though it changes the pH of water, it makes the water more basic. Alkaline water is not as dangerous as acidic water to marine life as some marine lives can adapt in alkaline water but die in acidic water. Also, with treatment, the pH of the water can be reversed to a more suitable pH before deposited into water sources.

The organic demulsifier does not make the waters acidic. Acidic water harm marine lives very greatly. It thickens mucus on gills of fishes which prevents the fishes from obtaining oxygen hence causing them to die. Acidic water also retards the growth of other marine lives like octopus and shrimps. The organic demulsifier formulates does not contribute to acidification of water which kills marine live, pollute the water, and prevent the growth of other sea weeds. The use of an organic demulsifiers instead of a synthetic demulsifier reduces the negative impacts these chemicals have on the environment..

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ЭКОНОМИЧНЫЙ ОРГАНИЧЕСКИЙ ДЕЭМУЛЬГАТОР НА ОСНОВЕ ЛИМОНЕНА

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АБСТРАКТ
Сырая нефть является наиболее важным продуктом в мире, поскольку позже она будет использоваться для производства нефти. На протяжении многих лет нефтяная промышленность всегда сталкивалась с проблемой добычи нефти, связанной с добычей нефти и воды. Вода, обнаруживаемая при добыче нефти, может быть обнаружена в двух формах: либо в свободной воде, либо в форме эмульсии. Это исследование проводится с целью изучить и предложить решение по деэмульгации воды при добыче нефти. Лимонен используется в процессе деэмульгирования. Лимонен получают из кожуры цитрусовых в качестве экологически чистого деэмульгатора. Для повышения эффективности к лимонену добавляются дополнительные агенты. Результаты этого исследования показывают, что органические деэмульгаторы эффективно разделяют эмульсии. В этом исследовании особое внимание уделялось органическим деэмульгаторам на основе лимонена как решению для отделения воды в масляной эмульсии. Это позволит добыче сырой нефти обеспечить как операционную эффективность, так и экологическую устойчивость.
Ключевые слова: сырая нефть, нефть, эмульсия, деэмульгаторы, нефтедобыча, лимонен, свободная вода.

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XÜLASƏ
Xam neftlər dünyanın ən vacib məhsullarıdır, çünki daha sonra neft hasil etmək üçün istifadə ediləcəkdir. Uzun illərdir ki, neft sənayesi həmişə neft hasilatında problemlə üzləşmişdir ki, bu da neft hasilatının su ilə müşayiət olunmasıdır. Neft hasilatında aşkar edilən su sərbəst su və ya emulsiya şəklində olan iki formada aşkar edilə bilər. Bu tədqiqat neft hasilatından suyun demulsifikasiyası üzrə həll yolunun araşdırılması və təklif edilməsi üçün aparılır. Demulsifikasiya prosesində limonen istifadə olunur. Limonen sitrus meyvələrinin qabıqlarından ekoloji cəhətdən təmiz demulsiya kimi alınır. Effektivliyi artırmaq üçün limonene əlavə agentlər əlavə edilir. Bu

araşdırmanın nəticəsi üzvi demulsiyaların emulsiyaları effektiv şəkildə ayırdığını göstərir. Bu tədqiqat neft emulsiyasında suyun ayrılması üçün bir həll kimi limonen əsaslı üzvi demulsifikasiyaları vurğulamışdır. Bu, xam neft hasilatına həm əməliyyat səmərəliliyini, həm də ekoloji dayanıqlığı təmin etməyə imkan verəcək.

Açar sözlər: xam neft, neft, emulsiya, demulsiya, neft hasilatı, Limonen, sərbəst su.