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Application of Microwave for Core Plug Cleaning

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Abstract

Rock extraction is a particularly major step for the preparation of rock samples for core analysis, it can be performed by many traditional techniques like Soxhlet, centrifuge, and flooding system. In this study, a new technique was suggested for core cleaning by microwave. New experiments were conducted as part of this research project, focusing on the cleaning of several types of core plugs under different conditions such as temperature and solvent volume. Additionally, the goal was to extract hydrocarbons from naturally saturated reservoir rocks that had been prepared for standard testing. These findings were then compared with the conventional method of cleaning samples using an organic solvent, specifically toluene.

The experiments successfully employed microwave technology for the cleaning process, effectively optimizing key variables including time, temperature, energy, and solvent quantity. The operational duration was set at 3 hours per day, using 20 ml of solvent, following numerous trials and iterations. This method allowed for the extraction of crude oil from 36 plug samples while achieving complete cleanliness of the rocks. Conducting porosity tests before and after the cleaning procedures showed the absence of any damage to the rock samples. Notably, there was an observed alteration in granular size within an acceptable range of 0.02 to 0.6. Further analyses were conducted, including gas chromatography and infrared analysis, both of which indicated that microwaves had no adverse effect on the crude oil extracted. Conclusively, the use of microwaves in the cleaning of rocks appears as a harmless, economically viable, environmentally friendly technique. The feasibility of this method was proved at the laboratory level, showcasing its potential for wider application.

Keywords: Core plugs, solvent, Microwave, Crude oil, cleaning.

استخدام الموجات الدقيقة في تنظيف السدادات الصخرية

الخلاصة:

ان استخلاص النفط من الصخور يعد خطوة اساسية لأعداد العينات الصخرية للتحاليل المكمنية للباب الصخري. حيث يتم اجرائها بأستخدام منظومة السوكسوليت و الطرد المركزي بالاضافة الى منظومة ضخ السوائل. في هذه الدراسة تم اقتراح تقنية جديدة مختلفة وهي استخدام الموجات المايكروية الدقيقة. لذا تم اجراء عدة تجارب كجزء من متطلبات البحث شملت تنظيف عدة انواع من العينات الصخرية تحت مختلف الظروف المختبرية مثل درجة الحرارة و نوع وكمية المذيب العضوي المستخدم. بالاضافة لذلك، فأن الهدف الرئيسي كان استخلاص الهيدروكربون من العينات الصخرية المكمنية المشبعة بشكل طبيعي بالنفط و المهيئة للتحاليل المكمنية القياسية. تم مقارنة نتائج هذه الطريقة مع الطرق التقليدية لتنظيف اللباب بأستخدام مذيبات عضوية ابرزها التولوين.

تم توظيف تقنية الموجات المايكروية الدقيقة بشكل صحيح وفعال لعملية التنظيف بحيث ساهمت بتعظيم العديد من المتغيرات الاساسية مثل وقت التجربة، درجة الحرارة المستخدمة، استهلاك الطاقة و كمية المذيب المستخدم. الوقت المستغرق لكل تجربة كان بواقع ثلاث ساعات تشغيل يومياً بأستخدام 20 ملليتر من المذيب و على عدة مراحل.

خلال هذه الطريقة تم استخلاص النفط من 36 سدادة صخرية وبشكل تام. وعند تنفيذ فحوصات حساب المسامية قبل وبعد عملية تنظيف اللباب اظهرت عدم وجود اي تضرر في العينات الصخرية. لكن كان هناك تشوه ملحوظ في الصخور ذات الحجم الحبيبي 0.6-0.02 ملم. واجريت تحاليل اضافية مثل تحليل كروماتوغرافيا الغاز و الاشعة تحت الحمراء والذان اظهرا ان الموجات الدقيقة ليس لديها اي تاثير على النفط الخام المستخلص. بشكل عام اظهرت الدراسة ان استخدام الموجات الدقيقة المايكروية في تنظيف الصخور لا تؤثر على الصخرة و بكفاءة اعلى، و انها تقنية صديقة للبيئة كما انها اكثر جدوى اقتصادية، اثبات جدواها اقتصاديا مختبريا يتيح لها احتمالية ان تستخدم على نطاق اوسع.

1. Introduction:

Sample plugs are drilled and trimmed 1-1.5in diameter and 3in long. Plugs are taken at regular intervals (often every 25 cm), parallel to layer planes for horizontal permeability. Plugs are cleaned by alternate extraction with hot toluene and methanol in Soxhlet extractors until no further discoloration of solvent occurs. This may take a long time depending upon permeability. Low permeability plugs are rarely completely free of residual brine and oil at this stage. There are several methods of cleaning the core. The actual method used will depend upon the properties of the core[1]

Rock extraction is one of the most important processes to prepare samples for reservoir core analyses, but this method consumes a large quantity of solvents and time which means excessive cost; so, it is necessary to innovate new high-efficiency ways to replace the traditional methods. There are many techniques for cleaning rock from organic compounds. Physical technique is based on a difference in solubility between the organic and inorganic phases and chemical technique is based on differences in reactivity towards oxidation[2]. The conventional techniques involved Soxhlet, shake-flask, and sonication extractions. Some more recently developed, more technically demanding methods include supercritical fluid extraction, pressurized liquid extraction, and matrix solid-phase dispersion. These are less common because of equipment costs, method development time, and higher risk of damage to the inorganic material because they all use a combination of elevated temperature and pressure[3].

Microwaves have been investigated during the past 50 years in laboratory and field tests to heat rocks and minerals. The main purpose is to assist mechanical methods for breakage, cutting, and comminution.

The primary responsibility of heat generation in microwaves is electricity. The molecules interacted with two different modes: dipolar rotation and ionic conduction. In dipolar rotation, a molecule continuously oscillates back and forth to align its dipole with the constantly changing electric field[4]. This rotational movement creates friction between the molecules, leading to the generation of heat. In ionic conduction, a free ion or ionic species moves in a straight line through space, also trying to align with the electric field fluctuations. Similar to dipolar rotation, the movement of these ionic species generates heat through friction. The efficiency of energy transfers increases with higher temperatures in both cases. Furthermore, the rate of heat generation is more efficient for species that are more polar and/or ionic[5].

In earlier work, detected a suitable solvent for the extraction of crude oil from carbonate rock and the performance of cleaning by microwave was investigated, where the time consumed was found to be 25% less than the conventional method for rock sample crude oil extraction[6].

This technique succeeds for that purpose, in this study a little modification performed on that procedure is using organic solvents in the experiments to dissolve pore-filling hydrocarbon. So different experiments were performed at different conditions to detect the best conditions for rock sample cleaning.

2. Experimental Works

2.1 Materials

Toluene purity 99%, Dielectric constant 2.4 Vapor pressure (atm) 0.197 (Sigma Aldrich), carbonate rock from Najma reservoir, sandstone from Zuber reservoir, and crude oil API 38.

2.2 Instruments

Microwave Mars (iwave 6), FTIR, Gc-simulated, and Soxhlet.

2.3 Sample Preparation

Two rock types used for the experiments carbonate and sandstone, underwent meticulous preparation involving the assessment of their weight, porosity, and dimensions. Subsequently, the samples are saturated with crude oil by the Vacuum Process illustrated in Figure (1). Following saturation, the specimens' weights were measured again to find the efficiency of the cleaning.

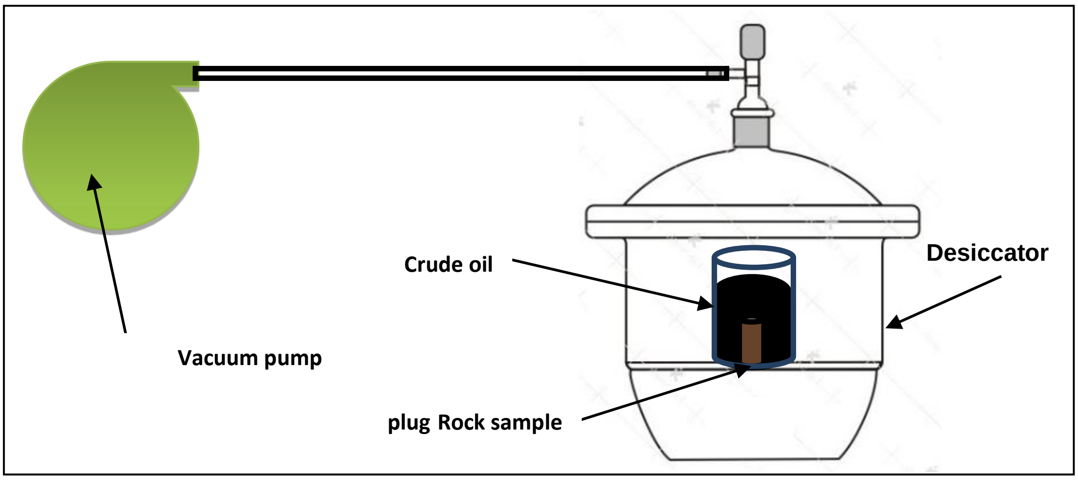

Fig. (1): Saturation vacuum process

2.4 Cleaning by microwave

The samples were carefully put within the instrument's cells, and with each cell a sample was submerged in (20) toluene. A glass holder was used for the sample inside the cell to prove efficient cleaning, considering that crude oil tends to accumulate in the cell bottom. Consequently, this prevents the recirculation of extracted crude oil back into the rock sample.

as shown in Figure (2). This insert comprised. Notably, its dimensions measured 1 inch in diameter, and 2 cm in height, and contained 19 holes, each with a diameter of 2 mm.

Fig. (2): Glass holder

Microwave experiment performed at a temperature of 100°C and a power output of 800 watts. The experiment continued until the solvent's color dissipated and became transparent, which indicates it became clean. Subsequently, a porosity assessment was conducted to clarify the effect on the plug that resulted from the two techniques.

2.5 Soxhlet cleaning

In the conventional method, a grouping of three samples is accommodated within a single Soxhlet apparatus. Here, one litre of toluene was used, and the soxhlet was operated at 110 °C. The operation continues until the toluene reaches a colourless state.

2.6 Effect of temperature

The effect of temperature on the efficiency of microwave-based cleaning was applied by subjecting carbonate and sand samples to various temperatures (60°C, 80°C, and 100°C). Following each temperature experiment, porosity measurements were conducted.

3. Results and Discussion

3.1 Microwave effect

When rock samples are irradiated by microwave, it will be affected by the controlled application of microwave power leading to a controlled temperature rising within the sample range. This process effectively cleans the samples without causing any significant alterations, except for minimal effects. In cases involving fractured samples, the extent of damage observed remained notably lower compared to the damage resulting from conventional cleaning methods. Consequently, this method exhibited superior efficiency.

A notable criterion for confirming the comprehensive cleaning of samples within the laboratory was the stay of toluene surrounding the sample. Furthermore, the weight of each sample, post-treatment, compared with its weight before saturation. Some of the samples showed slight weight reduction after cleaning in comparison to their weight before crude oil saturation. This phenomenon can be attributed to the minor loss of granules in these rock samples. However, this loss was generally less than what is observed with the traditional cleaning method. In traditional cleaning, the accumulation of rock granules often results in the bottom of a round flask, whereas the microwave instrument's cells remain free of such deposits.

Figure (3) shows the change in sample weight of four plugs that were irradiated with microwaves in the presence of solvent. The reduction in weight after the experiment was measured to ensure the absence of any granules or clay. The recorded weight difference was a mere 0.071 g.

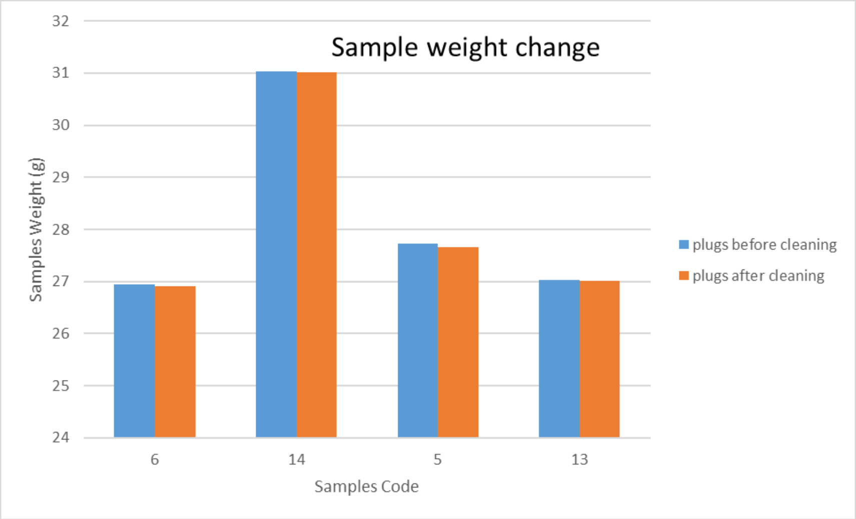

Fig. (3): weight of plugs sample before and after cleaning by microwave

3.2 Techniques Comparison Cleaning for carbonate rock:

The comparison between microwaves and traditional methods of cleaning. The process of heating mineral components within rocks during microwave treatment can result in volumetric expansion. This expansion induces stress along grain edges, potentially leading to intergranular cracks that might weaken the rock structure if high power levels of 800 watts are employed [8]. Consequently, a cautious approach was adopted during the study, utilizing a power of 800 watts evenly distributed among eight samples, translating to 100 watts per sample.

An interesting observation emerged when analyzing limestone rock samples. Swelling was observed in one of these samples, a phenomenon attributed to the elevated sulfur content in the crude oil. The presence of hydrogen sulfide resulted in high vapor pressure, particularly under the temperature of 100°C. This combination facilitated increased sample expansion.

A comparison between the two methods involved the measurement of pore volume in rock cleaning as shown in Figure (4). No significant differences were detected between the microwave and traditional methods. This suggests that microwave technology did not adversely affect the rocks, substantiated by the convergence of results from the two methods, wherein differences ranged within the interval of 0.005 to 0.2. It's worth noting that the gradual cooling of the samples contributed to minimizing post-cooling size variations.

Furthermore, porosity measurements: low-porosity samples show more evident changes in pore volume compared to high-porosity samples. It may be that minor cracking in low-porosity samples can create new pores. In contrast, expansion-induced changes affect high-porosity samples differently the vapor pressure diminished through pore spaces.

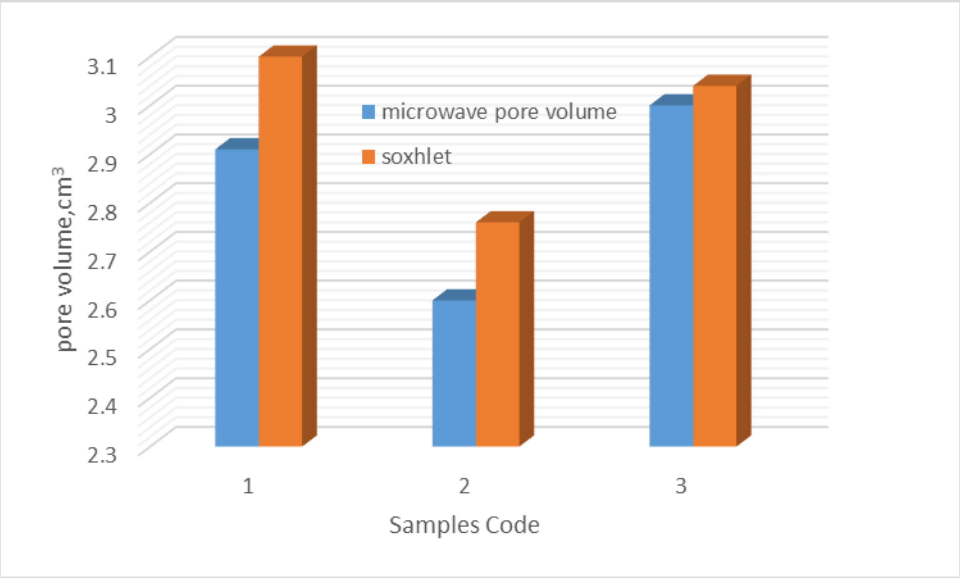

Fig. (4): Pore volume comparison for cleaning plugs by microwave and Soxhlet

Sandstone rocks impregnated with crude oil have demonstrated the ability to absorb microwaves, attaining the designated temperature efficiently. This absorption mechanism mirrors the behaviour observed when subjecting carbonite rock samples to microwave exposure.

3.3 Effect of Microwaves on Sandstone:

A comprehensive examination of microwaves' impact on sandstone rock samples was conducted. These sand samples were radiated by a microwave apparatus, with specific operational parameters of 60 ºC and 800 watts.

The assessment of pore size variations within the samples was measured. Certain samples showed sufficient increases in pore size, ranging from 0.6 to 0.9, while others demonstrated relatively minor size increments, shifting from 1.7 to 1.8 as shown in Figure (5). Despite these modest fluctuations, the integrity of the rock samples remained unchanged.

Sandstone rocks characterized by a coarser grain size showcased expedited hydrocarbon material extraction. This phenomenon can be attributed to the rock's elevated permeability, due to increasing void volume that increased spacing between grains.

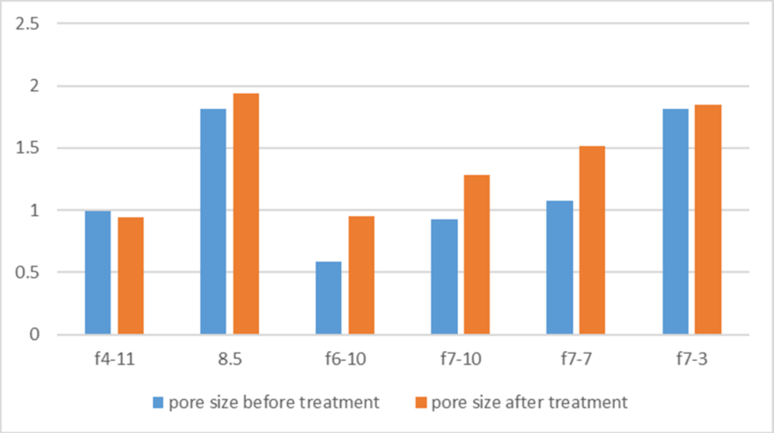

Fig. (5): Pore size before and after microwave treatment.

3.4 Techniques comparison for cleaning Sandstone

In collaboration with the Basra Oil Company, an investigation was undertaken involving naturally saturated samples. These samples were divided into two sections for cleaning purposes, employing both the conventional method at 110 degrees Celsius and the microwave method under operational conditions of 60 degrees Celsius and 800 watts. The primary objective was to highlight the disparities between the two methods in terms of sand sample preservation and their impact on porosity assessment. Porosity analyses proved to be a precise benchmark for evaluating the efficacy of microwaves, particularly for cleaning naturally impregnated rocks.

When employing sandstone with constant quantities of toluene solvent, it becomes evident that microwave utilization led to reduced cleaning time. Variations in operating conditions between the microwave method and the traditional approach were observed, that the distinctive characteristics of sandstone that necessitated lower heat levels due to its comparatively lesser cohesion.

In the context of routine cleaning via the Soxhlet method, instances of sample damage were noted, although these occurrences were notably less frequent with the microwave technique. The microwave approach also demonstrated a reduction in solvent consumption of approximately 45% in comparison to the traditional method. Specifically, the microwave method incurred a minimal solvent loss of 5%, while the traditional method experienced a substantial 50% loss due to evaporation, a comparison shown in Figure (6). For heavily crude oil-contaminated samples, one past half litres of solvent was employed using the microwave method, whereas two litres were required for the traditional method. This efficiency gain can be attributed to the hermetic sealing of the microwave device cells.

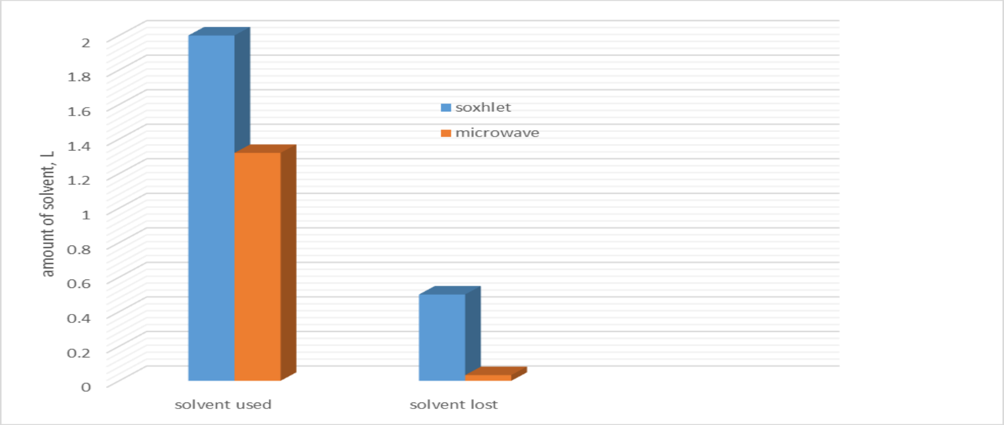

Fig. (6): Comparison between the two methods of the amount of solvent lost during the experiments.

Figure (7) emphasizes the shorter cleaning duration associated with the microwave method, reflecting its efficiency. This efficiency can be attributed to several factors, including the solvent's behaviour, the indirect impact of microwaves on the sample, and the water content within the rock. Water content significantly contributes to elevating internal rock temperature, generating steam pressure that aids in expelling oil**.**

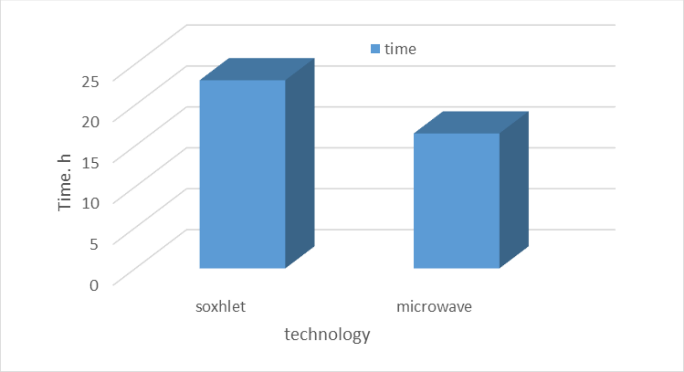

Fig. (7): A comparison between the two methods for the time required to complete the cleaning.

An important distinction lies in the cooling mechanisms: the conventional extraction system necessitates water cooling, which consumes water and ethanol, to condense the solvent vapour and minimize losses. In contrast, the microwave device employs an efficient ventilation system and vacuum mechanisms, eliminating the need for extensive water consumption and contributing to environmental sustainability.

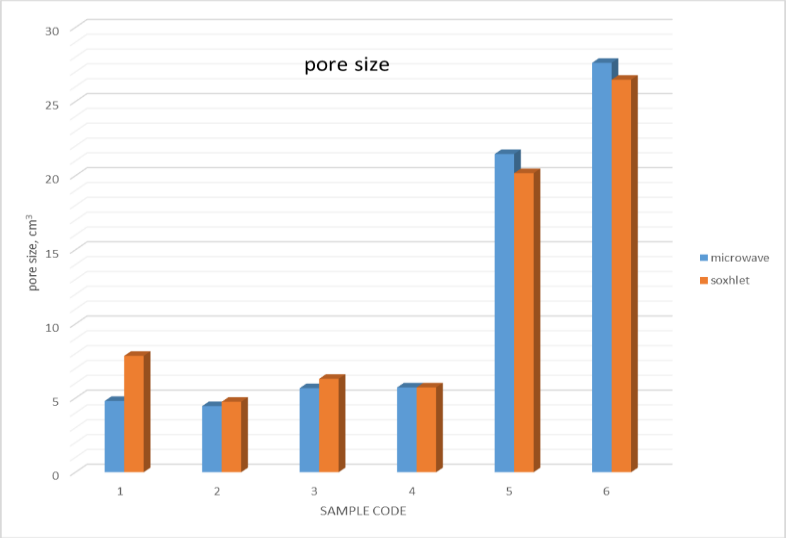

Fig. (8): Comparison of pore size between cleaning by microwave and soxhlet

3.5 Temperature effect for sandstone and carbonate rocks.

Figure (9) shows the variance in rock pore volume attributed to higher temperatures in the traditional method (reaching 110 degrees Celsius). This elevated temperature weakens granular bonding, leading to fragmentation, unlike the uniform and gradual thermal distribution achieved by the microwave, further evidenced by the weight change of the sample.

Through rigorous experiments, optimal conditions were derived for treating different rock samples (carbonate and sandstone) at varying temperatures. By preparing and subsequently cleaning the samples using microwave technology at temperatures of 60, 80, and 100 degrees Celsius and 800 watts, specific porosity measurements were obtained to understand wave impact, a crucial parameter given the reservoir's importance in oil storage calculations.

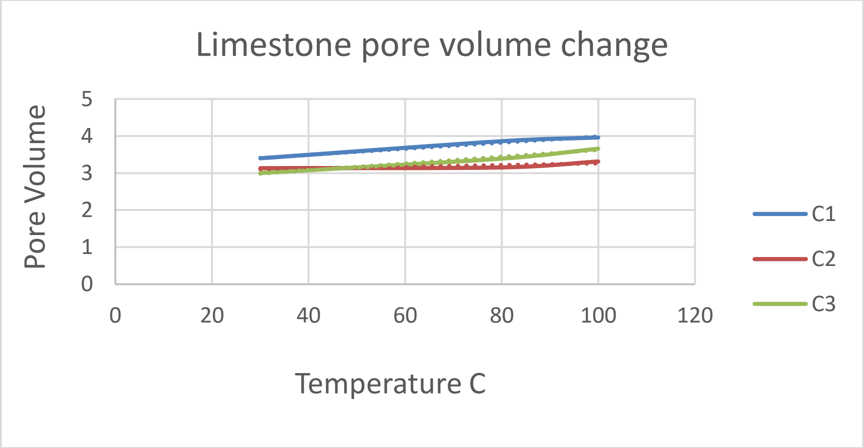

Fig. (9): Effect of temperature on pore volume for limestone samples

Moreover, when applying the microwave method to sandstone with consistent toluene solvent quantities but different time durations, a noticeable reduction in cleaning time was evident, echoing findings observed with carbonate rocks as shown in Figure (10).

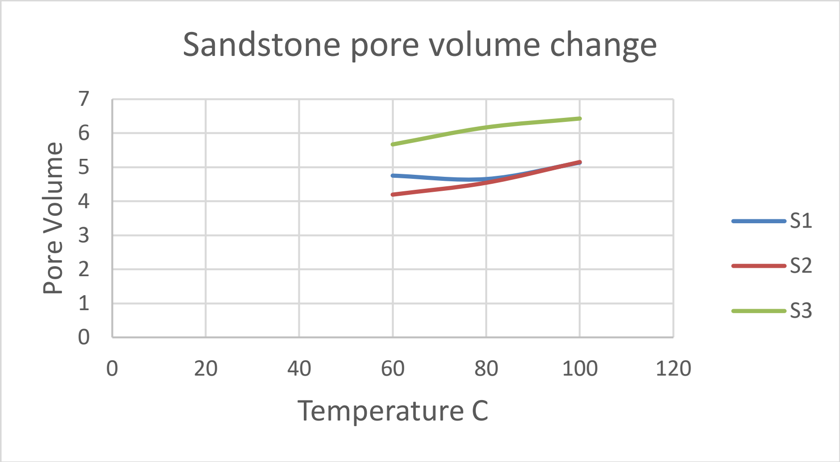

Fig. (10): Effect of temperature on pore volume for sand samples

Figures (9 and 10) underscored the consistent behaviour of curves, indicating a direct relationship between temperature and changes in pore size. However, slight deviations were attributed to varying mineral content, such as binder and clay mineral ratios. Notably, certain clay minerals lose water content at temperatures exceeding 65°C. Additionally, discrepancies in rock texture, pore shape, and size contributed to variations within the curves.

3.6 Fourier Transformation FTIR check

Comprehensive evaluations were executed using an infrared device throughout experiments exposing various samples to microwaves. This meticulous analysis revealed a crucial insight: Microwaves exhibited no discernible impact on the properties of crude oil. This conclusion is graphically depicted in Figures (11) and (12).

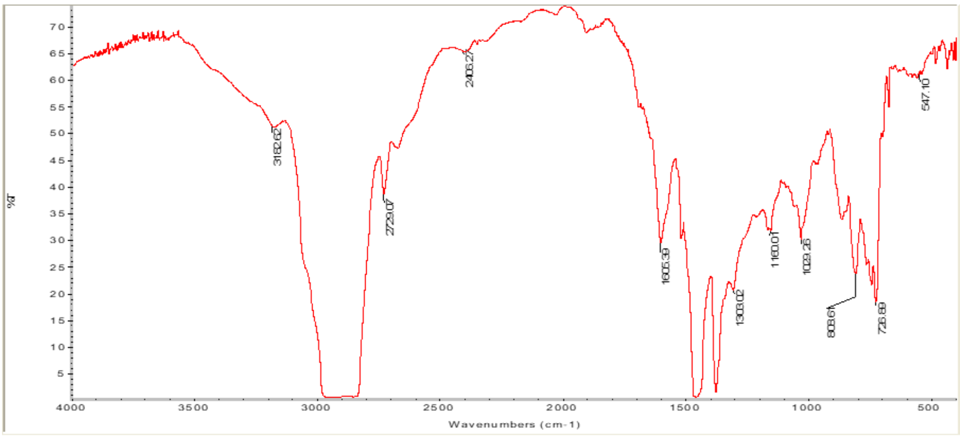

Fig. (11): FTIR spectrum for west qurna crude oil before microwave treating.

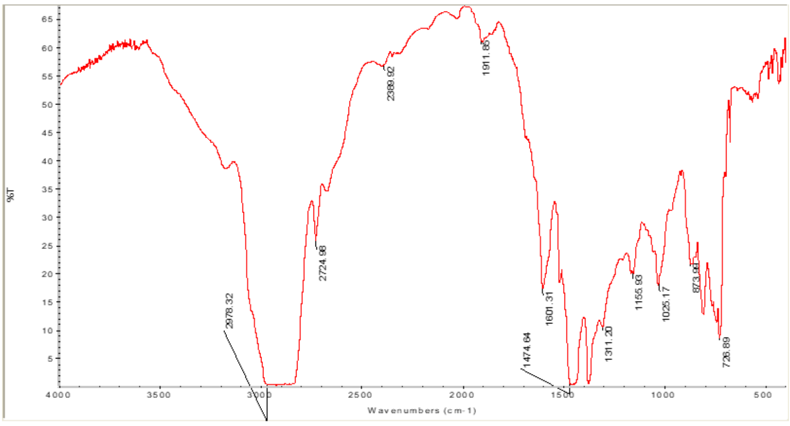

Fig. (12): FTIR spectrum for west qurna crude oil after microwave treatment.

3.7 Check GC Simulated

Numerous assessments were performed on crude oil before and after exposure to microwaves. The outcomes consistently conveyed an important observation: the inherent characteristics of the crude oil remained unaffected by microwave exposure. This conclusion finds validation in the results obtained through gas chromatography (simulating distillation) using the GC-Simulated technique, depicted in Figures (13) and (14). These figures portray a graphical representation, wherein the horizontal axis corresponds to the percentage of hydrocarbon compounds, while the vertical axis corresponds to the boiling points of these compounds. These tests collectively underscore the preservation of crude oil properties despite exposure to microwaves.

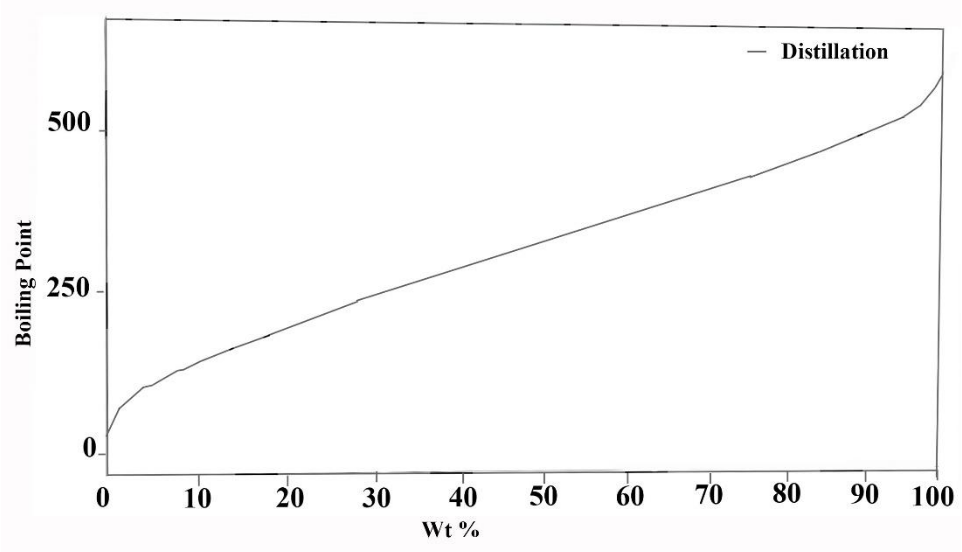

Fig. (13): GC-Simulated for west qurna crude oil before microwave treatment. :() Figure

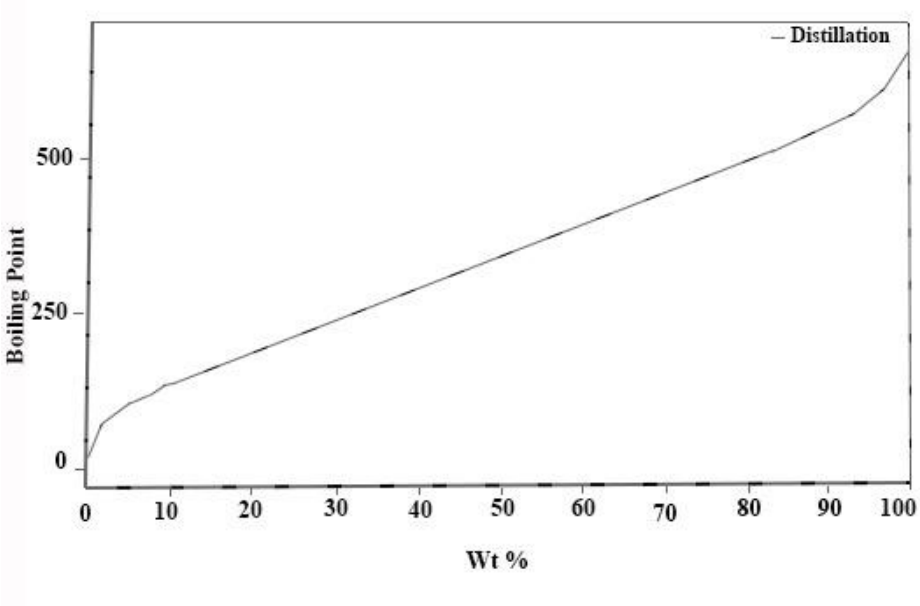

Figure (14): GC-Simulated for west qurna after microwave treatment

3.8 Economic feasibility

Demonstrating the Method's Economic Viability:

An essential facet in substantiating the method's efficiency is its economic feasibility, an aspect succinctly highlighted in Table (1). Notably, the traditional method necessitates double the energy requirement compared to the microwave method. Additionally, various technical considerations come into play, including cooling mechanisms and additional spatial requirements, particularly for optimal laboratory ventilation efficiency. Moreover, the traditional method incurs double the solvent consumption in comparison to the microwave method. This confluence of factors solidifies the economic superiority of the microwave approach.

Table (1) shows the economic and technical feasibility of the comparison between the traditional method of cleaning Soxhlet and microwave oven.

	microwave	Soxhlet
ID/1lit oil extracted	36000	54000
Power consumption	15 Amp.	40Amp
Environmental hazard	Environmentally friendly (no emission)	Non-friendly (high emission
safety	More safety to use	less

4. Conclusions

In conclusion, the utilization of microwave technology for rock sample cleaning represents a significant advancement in the realm of petroleum operations. This technique has been successfully evaluated for cleaning oil-saturated rocks, yielding highly effective outcomes. A comparative analysis between the conventional and microwave methods reveals that the latter offers superior performance with less impact on samples, reduced energy consumption, and minimized solvent usage. Technology manages to preserve the properties of both rocks and crude oil without compromising them, while also saving time and resources.

Furthermore, the study demonstrates that microwave technology is economically viable in comparison to traditional approaches. Lower energy consumption reduced solvent usage, and other associated resource savings can potentially enhance the overall cost-effectiveness of cleaning and extraction processes in the oil and gas industry. The combination of preserving rock and crude oil properties, coupled with improved economic and environmental efficiency, underscores the pivotal advantages of microwave technology in this context.

Based on the achieved results, it can be confidently stated that microwave technology presents a promising step toward enhancing rock cleaning and extraction processes in the oil and gas industry. This is achieved by saving time, effort, and specific resources while maintaining the quality of materials involved in these processes.

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