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The utilization of nanoparticles to modify surface properties in acidification processes

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Abstract

Stimulation plays a crucial role in the oil and gas industry, particularly in the acidizing process. Thus, the development of acidification processes is essential for improving the productivity index and enhancing the physical properties of reservoirs. This research investigates the effect of Tin oxide (SnO_2) and Titanium dioxide (TiO_2) nanomaterials on the surface properties in acidic media of hydrochloric acid (HCl) at various acid and nanomaterials concentrations, as well as their impact on the wettability of carbonate rocks. To achieve this, surface tension, contact angle, and zeta-potential were measured using various instruments. The findings show that SnO_2 and TiO_2, at a concentration of 1000 ppm, significantly reduce surface tension to 34.69 and 43.44 mN/m, respectively, while maintaining stability with zeta potential values up to 51.27 and 49.17 mV, respectively. These results exceed the generally accepted stability threshold of around ±30 mV, indicating a strong electrostatic repulsion that prevents agglomeration and ensures prolonged suspension stability. Additionally, the findings show a substantial decrease in contact angles, from 95.17° to 16.32° with SnO_2 and from 98.48° to 25.26° with TiO_2. This shift indicates a transition from non-wetting to a more wetting condition, which is crucial because it reflects an increased affinity of the acid solution for the carbonate rock surfaces. This results in promoting better acid distribution and enhancing rock dissolution. In conclusion, both SnO_2 and TiO_2 reveal excellent performance as surfactants in the stimulation process, presenting substantial benefits for the acidizing process in the oil and gas industry.

Keywords: Acidic Nanofluids; Surface Properties; Zeta Potential Stability; Contact Angle of Nanofluids; SnO_2 nanoparticles; TiO_2 nanoparticles.

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1- Introduction

Nanofluids are sophisticated colloidal suspensions consisting of nanoparticles, which are typically less than 100 nm in size, dispersed in a base fluid. The nanoparticles can be metals, metal oxides, or other compounds, and the base fluids can range from water to various chemical solutions. Nanofluids exhibit improved physical and chemical properties compared to base fluids alone, making them highly effective in a variety of industrial applications [1-3].

Highly acidic solutions, particularly those with strong acids like hydrochloric acid (HCl), are widely used in various industrial processes. In the oil and gas industry, highly concentrated HCl is employed in well stimulation techniques such as acidizing, where it helps dissolve mineral deposits in the rock formations to enhance oil and gas flow. Other applications include metal etching, pH control, and chemical synthesis [4-6].

Integrating nanofluids into highly acidic solutions like concentrated HCl can significantly enhance the performance and efficiency of the acid treatment. This integration leverages the unique properties of nanoparticles to overcome some of the limitations of conventional acid treatments [7].

Surface tension in nanofluids containing highly acidic solutions like HCl can be influenced by nanoparticles in several ways. The presence of nanoparticles, such as SiO_2, Al_2O_3, and ZnO, in hybrid nanoparticle-acid fluids (HNAFs) has been shown to impact interfacial tension (IFT) by reducing it with increasing acid concentration for SiO_2 and Al_2O_3, while showing a reverse trend for ZnO. Additionally, the wetting behaviour of Nano-droplets can be affected by the interaction between the droplets and the substrate, with the contact angle decreasing with increased ethanol concentration and solid-liquid interaction [8, 9].

High zeta potential values play a crucial role in enhancing the stability, whether positive or negative, of nanofluids by promoting strong electrostatic repulsion between particles, as a result, it prevents aggregation and maintains a stable dispersion [10-12]. The contribution to a stable dispersion that is essential for preserving the uniformity and effectiveness of the fluid is a consequence of the presence of nanoparticles in HCl, which can lead to the acquisition of charges [13]. Understanding and

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controlling factors like nanoparticle concentration ratio, base fluid type, and size are key to achieving stable nanofluids with enhanced thermos-physical properties [14].

Altering the wettability of formation plays a crucial role in impacting enhanced oil recovery (EOR) processes, therefore, nanoparticles are considered to be a highly effective addition due to the significant impact in altering the wettability of formation rock, transitioning it from oil-wet to water-wet. Studies have shown that nanoparticles like TiO_2 and ZnO can have the ability to shift surfaces from oil-wet to water-wet conditions [15, 16].

The size and concentration of nanoparticles are vital factors for influencing wettability alteration and oil recovery, in which smaller particle sizes and optimal concentrations lead to enhanced wettability changes and increased oil recovery rates [17, 18]. Additionally, the surface-modified TiO_2 nanoparticles revealed a significant alteration in rock wettability, as higher nanoparticle concentrations would lead to higher wettability changes [19].

Furthermore, the addition of TiO_2 nanoparticles to acidic solutions can alter the zeta potential of the solution, increasing its stability due to a change in the electrostatic potential at the particle surface [18]. Besides that, the surface tension of acidic solutions is reduced when they contain TiO_2 nanoparticles, leading to enhanced wettability of carbonate rocks by making them more hydrophilic [20, 21].

Also, the contact angle of carbonic rock is decreased by TiO_2 nanoparticles when added to acidic solutions, referring to an increment in the hydrophilicity properties and a rise in wettability of the surface [9], [19]. In acidic media, tin oxide (SnO_2) exhibits significant interactions, affecting surface tension, contact angle, and zeta potential [22].

The primary aim of this research is to investigate the behaviour of nanoparticles when exposed to strong acidic environments, with a focus on evaluating their compatibility and effectiveness. By understanding how nanoparticles interact in such conditions, the study seeks to uncover their potential for improving industrial applications.

Given the significant potential of nanoparticles to modify surface physical properties, this research emphasizes their promising role in advancing acidizing processes within the oil industry. By considering these effects, the study aims to provide valuable insights that could lead to the development of more efficient and effective acidizing techniques, ultimately contributing to the optimization of oil extraction processes.

2- Materials and methods

Concentrated hydrochloric acid was diluted from 37.6% to a concentration of 15% by adding distilled water. Then, the previously purchased nanoparticles were added in different weight ratios to the diluted solution with a volume of 100 ml. As mentioned in Table 1 and Table 2, which specify the HCL and nanoparticles concentration:

Table 1. Specification of concentrated HCl

Acid Name	Concentration (%)	Molecular Weight	Density(gm/cc)	Impurities Percentage (%)	Company Name
Hydrochloric Acid (HCl)	37%	36.46	1.18	<0.001%	Thomas Baker

Table 2. Specification of purchased nanoparticles

NP Name (Symbol)	Purity (%)	Particle Size (nm)	Colour	Morphology	Company Name
Titanium Oxide (TiO_2)	99.5%	20	White	Spherical	Skyspring Nanomaterial
Tin Oxide (SnO_2)	99.9%	60	White	-	Skyspring Nanomaterial

After completing the preparation of each sample, it is placed in an ultrasonic vibrator to ensure the diffusion of nanoparticles within the highly concentrated acid solution. Tests were conducted on the surface tension between the solution and the air, the contact angle between the nanofluid without acid and the carbonate rock, as well as the zeta potential, as follows.

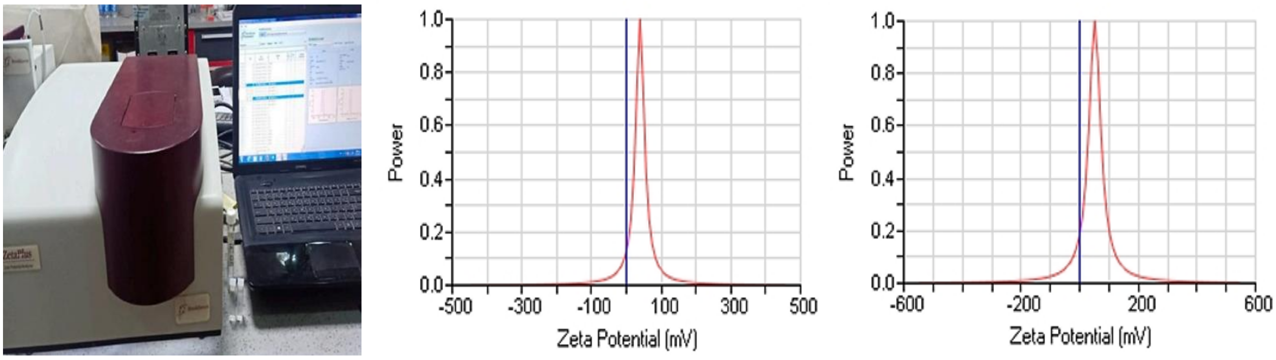
2.1. Surface tension

Measuring surface tension using the Force Tensiometer Sigma 703D involves a detailed and precise procedure. After ensuring all components, including the tensiometer, sample container, and probe are clean, four types of nanofluid were prepared; firstly, two solutions consist of distilled water and once with nanoparticles of (250, 500, 750, 1000 PPM) of SnO_2 and the other with (250, 500, 750, 1000 PPM) of TiO_2. Secondly, the other two types are with the existence of diluted HCl of a concentration of (3%, 6%, and 15%) with two types of nanoparticles of (500, 1000 ppm) for each SnO_2 and TiO_2.

Then, place the sample container with the nanofluid on the stage of the tensiometer. Slowly, the probe is lowered until it just touches the surface of the nanofluid. The instrument will begin to measure the force exerted by the nanofluid on the probe. The tensiometer will measure and record the force exerted on the probe. This force is directly related to the surface tension of the nanofluid.

2.2. Zeta potential

Measuring the zeta potential of a nanofluid using Brookhaven's ZetaPALS, as in Fig. 1, involves several precise steps. Firstly, all components, including the sample cell, electrodes, and any other accessories, are thoroughly cleaned to avoid contamination. Secondly, two types of Nanofluid were prepared in which consist of HCl concentration (1.5%, 6%, and 15%) with two types of nanoparticles of SnO_2 and TiO_2, and each one is within (500, 1000 PPM) concentration. Then the samples are well-dispersed and free from large agglomerates or bubbles. The Brookhaven ZetaPALS instrument is turned on and allowed to initialize.



(a) **(b)** **(c)**

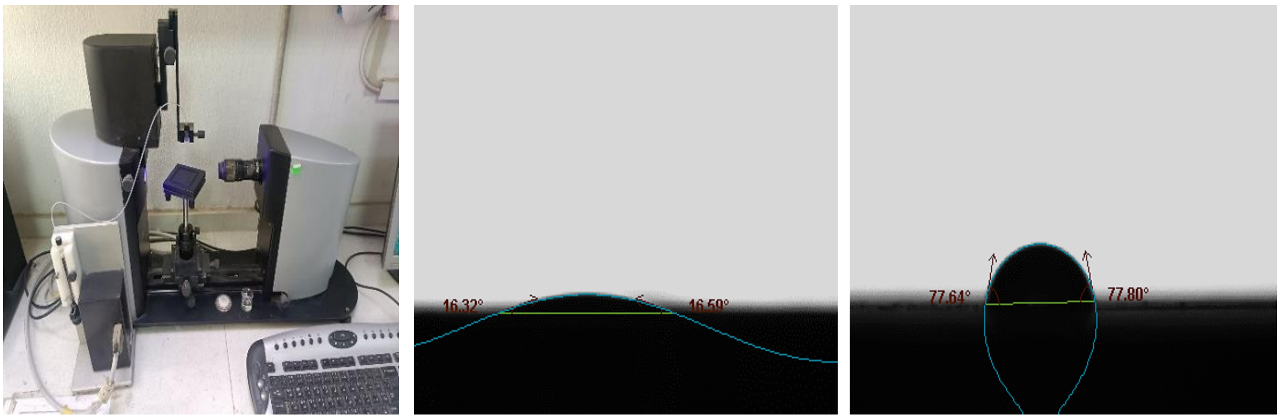
Fig. 1. The ZetaPALS device is represented with its results as follows: (a) ZetaPALS device with its program. (b) Result of SnO_2 of (1000 ppm) and HCl concentration of (1.5%). (c) Result of TiO_2 of (500 ppm) and HCl concentration of (15%)

Then the sample cell is filled with the nanofluid, and the electrodes in the sample cell are inserted. The measurement process via the software interface is initiated. The instrument will apply an electric field to the sample and measure the velocity of the nanoparticles. The ZetaPALS instrument uses Phase Analysis Light Scattering (PALS) to determine the electrophoretic mobility of the nanoparticles. The software will record the mobility data and calculate the zeta potential. The software will automatically calculate the zeta potential based on the measured electrophoretic mobility, using the Smoluchowski equation.

2.3. Contact angle

Attension Theta Optical Tensiometers, as illustrated in Fig. 2a, are sophisticated instruments designed to measure contact angles, providing valuable insights into surface properties and wettability. Ensure the tensiometer and its components, including the sample stage, syringe, and needles, are thoroughly clean to avoid contamination.

Plugs, which are cut from a carbonate rock core of the Yamama formation in the West-Qurna oilfield in southern Iraq, were used for measuring the contact angle with the nanofluid. Since the plugs cannot immerse in acidic solution due to the reactivity of HCl in the carbonate rocks, the nanofluid was only prepared from distilled water and a concentration of (500, 1000 PPM) of both SnO_2 and TiO_2 and the immersing period is considered to be (0, 30, 90, 180 min) for each sample. The surface on which the droplet will be placed is clean, smooth, and dry. The syringe is then filled with the nanofluids, ensuring it is free from bubbles.



(a) **(b)** **(c)**

Fig. 2. The Attension Theta Optical Tensiometers instrument is represented with its results as follows: (a) Attension Theta Optical Tensiometers instrument. (b) Result of SnO_2 of (1000 ppm) with an immersed period of (180 min). (c) Result of TiO_2 of (1000 ppm) with an immersed period of (30 min)

The filled syringe is attached to the syringe holder on the tensiometer. Adjust the stage height and ensure it is level. Slowly, a small droplet of the liquid is dispensed onto the surface of the plug. Using the tensiometer's camera to focus on the droplet. Capture an image of the droplet, as in Fig. 2(b and c), using the tensiometer's software interface. This image will be used to analyze the contact angle. This is typically done by fitting a curve to the droplet profile and calculating the angle at the contact points where the droplet meets the plug.

2.4. XRF measurement

To measure elements percentage through the SPECTRO XEPOS bench-top XRF, the rock sample must be prepared by grinding it into a fine powder to ensure homogeneity. Load the powdered sample into a sample holder. Press the sample into a pellet to ensure a flat surface. The SPECTRO XEPOS XRF spectrometer will start the analysis. The instrument will use a primary X-ray beam to excite the sample, which will then emit secondary X-rays characteristic of the elements present. The SPECTRO XEPOS will measure the intensity and energy of the secondary X-rays and calculate the elemental composition of the sample.

3- Results and discussion

The impact of SnO_2 and TiO_2 nanoparticles (NPs) on the solutions in varying concentrations for both NPs and HCl can be analysed based on the results as follows:

3.1. The surface tension (S.T.)

As shown in Fig. 3 demonstrating the results of surface tension for the distilled water and nanofluid for two types of nanoparticles and concentration without the effect of HCl. Increasing the concentration of SnO_2 nanoparticles reduces the surface tension. Where at 1000 ppm, the surface tension significantly drops from 73.2 mN/m to 50.04 mN/m. Same results for TiO_2 when increasing TiO_2 nanoparticle concentration reduces the surface tension, but not as dramatically as SnO_2. At 1000 ppm, the surface tension drops to 62.33 mN/m from 73.2 mN/m.

Whereas when the pH of the solution changed due to HCl addition at different concentrations, the difference is even getting better. Fig. 4 shows that the presence of HCl further reduces the surface tension. Higher HCl concentration leads to a lower surface tension. At 1000 ppm SnO_2 and 15% of HCl, the surface tension is lowest at 34.69 mN/m. Similar to SnO_2, the addition of HCl to TiO_2 nanoparticles reduces the surface tension. Therefore, higher HCl concentration correlates with lower surface tension.

Both SnO_2 and TiO_2 nanoparticles reduce the surface tension of the solution. SnO_2 nanoparticles have a more pronounced effect on reducing surface tension compared to TiO_2 nanoparticles. The addition of HCl further decreases the surface tension in both SnO_2 and TiO_2 systems.

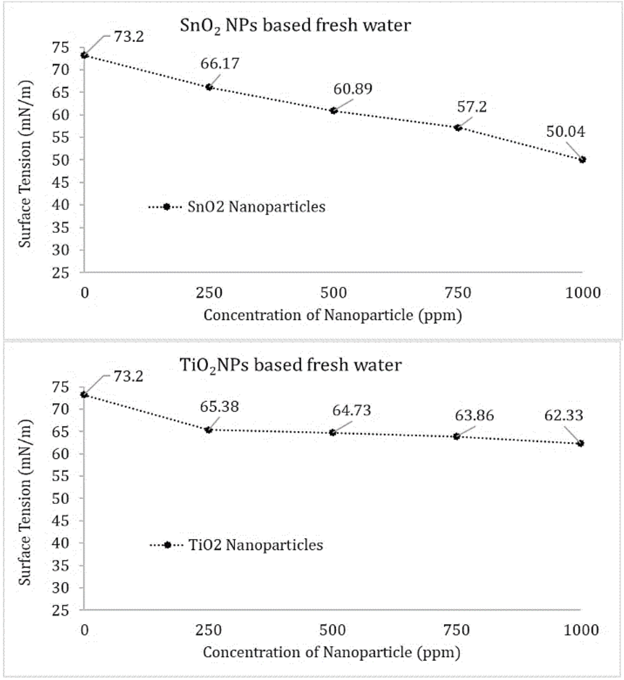


Fig. 3. Represents the effect of SnO_2 and TiO_2 nanofluid with distilled water on the surface tension at different concentrations

3.2. Zeta potential

The Results show the zeta potential measurements for SnO_2 and TiO_2 nanoparticles at different concentrations of HCl. As shown in Fig. 5 below it shows for both SnO_2 and TiO_2 nanoparticles, the zeta potential increases with increasing HCl concentration. This suggests that higher acidity stabilizes the nanoparticles more effectively, leading to higher zeta potential values. At 500 ppm and 1000 ppm, the zeta potential trends are similar. However, at lower HCl concentration, 500 ppm tends to show slightly higher zeta potential compared to 1000 ppm. Both SnO_2 and TiO_2 show an increase in zeta potential with increasing HCl concentration, but TiO_2 has higher zeta potential values compared to SnO_2 under the same conditions.

3.3. Contact angle measurements

For carbonate rock plugs, Yamama carbonate formation from West-Qurna oilfield in southern Iraq, the XRF shows that 92.87% of the rock is composed of Calcium component, referring to limestone as appeared in Fig. 6.

After immersion in nanofluids with different concentrations of SnO_2 and TiO_2 nanoparticles over various times. Higher concentrations as Fig. 7. below refers that (1000 ppm) lead to lower contact angles compared to lower concentrations (500 ppm), indicating increased hydrophilicity. Longer immersion times result in significantly reduced contact angles, indicating that the wettability of the carbonate rock surface increases over time.

The two types of nanoparticles show similar trends of decreasing contact angle with increasing concentration and immersion time. TiO_2 shows a slightly higher initial contact angle compared to SnO_2, but both reach similar low contact angles after 180 minutes of immersion.

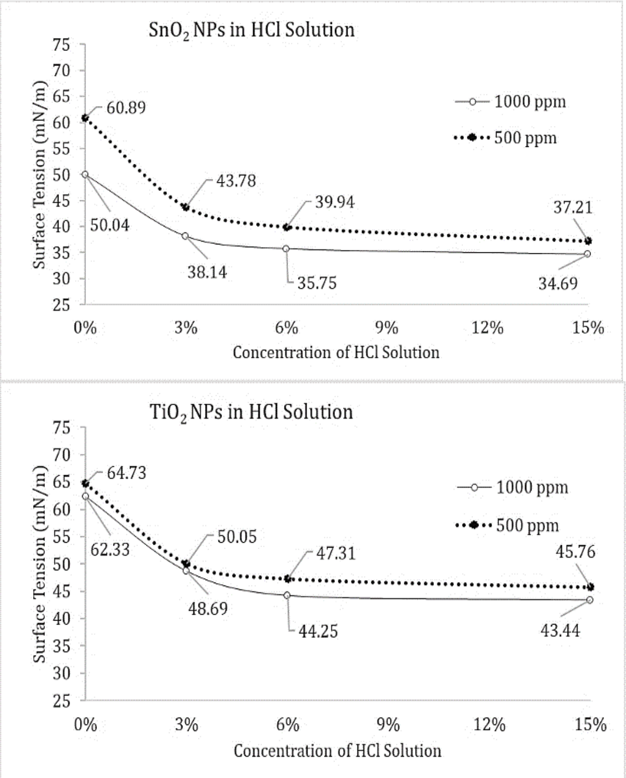


Fig. 4. Represents the surface tension of SnO_2 and TiO_2 nanofluid of (500 and 1000 ppm) with different HCl concentrations

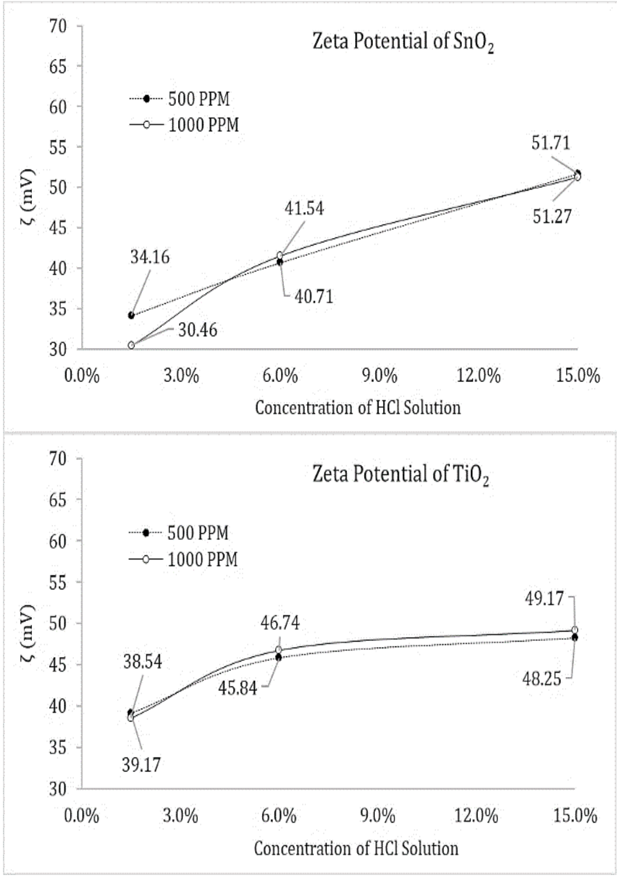


Fig. 5. Represents the zeta potential results of SnO_2 and TiO_2 nanofluid of (500 and 1000 ppm) with different HCl concentrations

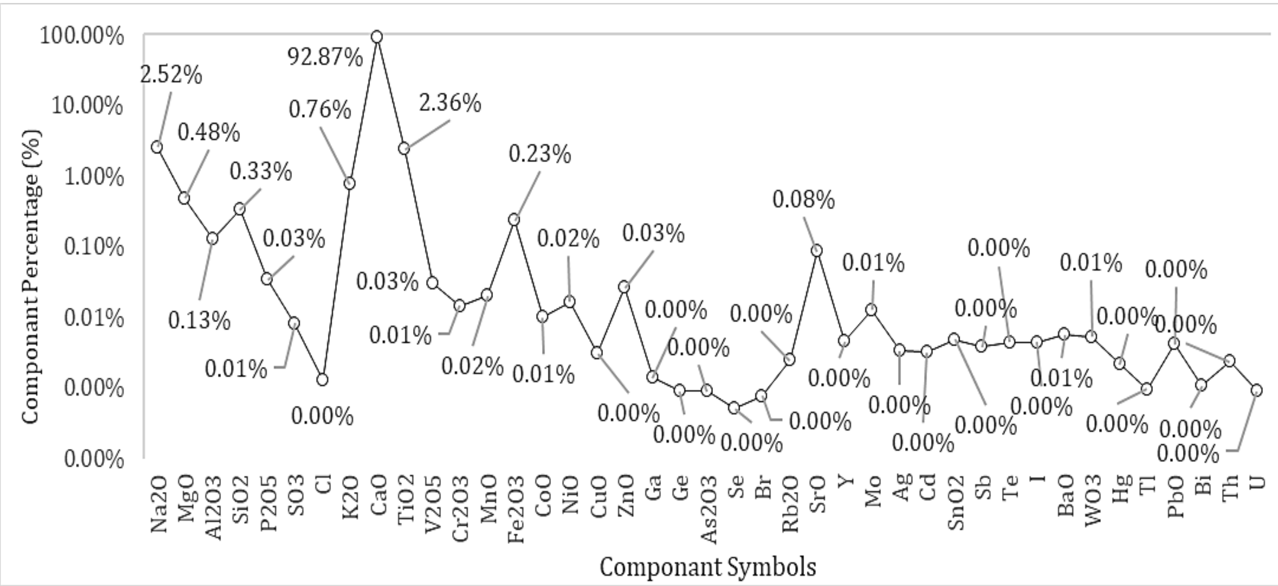


Fig. 6. Represent the XRF results for Yamama carbonate rock, West-Qurna oil field

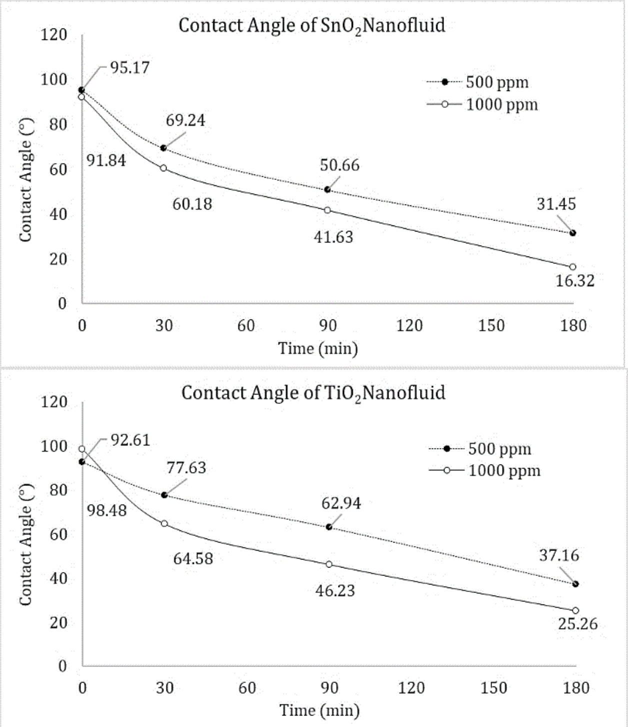


Fig. 7. Contact angle results for SnO_2 and TiO_2 nanofluids with different immersing periods

4- Conclusions

SnO_2 and TiO_2 nanoparticles, especially when combined with HCl, significantly reduce the surface tension of solutions. This reduction is crucial for applications involving surface interaction in acidic environments. The stability of these nanomaterials in solution is tested by zeta potentials, which measure 51.27 mV for SnO_2 and 49.17 mV for TiO_2. These potential values indicate that nanomaterials are well-dispersed and stable in acidic media, which is crucial for maintaining effective concentrations during the acidizing process. Enhanced suspension stability, as indicated by zeta potential, is essential for ensuring that these nanomaterials remain homogeneously distributed within the solution. Additionally, SnO_2 and TiO_2 nanoparticles significantly reduce the contact angle of carbonate rock, thus improving its wettability. These behaviours suggest that these nanoparticles can enhance the surface properties of acidic media and increase the wettability of carbonate rocks; SnO_2 and TiO_2 nanomaterials are promising additives for acidizing operations in the oil and gas industry. As a result, improve the physical and chemical properties of acid solutions beside enhance the contact interactions with carbonate reservoirs, making them highly effective in stimulating hydrocarbon production. It is essential to employ several types of nanoparticles, as research in this area is limited. Furthermore, petroleum operations necessitate a thorough understanding of these factors to facilitate their future implementation.

Conflict of interest statements

The authors declare that they have no conflict of interest.

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إستخدام الجسيمات النانوية لتعديل خصائص السطح في عمليات التحميض

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الخلاصة

التنشيط يلعب دورًا حيويًا في صناعة النفط والغاز، خاصة في عملية الانعاش، لذا فإن تطوير عمليات التحميض يُعد جانبًا مهمًا لتحسين مؤشر الإنتاجية وتعزيز الخصائص الفيزيائية للمكمن. في هذا البحث، تم الكشف عن تأثير المواد النانوية من أكسيد القصدير(SnO_2) وأكسيد التيتانيوم (TiO_2) على الخصائص السطحية في وسط حامضي من حمض الهيدروكلوريك (HCl) بتركيزات مختلفة لكل من الحمض والمواد النانوية، بالإضافة إلى تغير التبلل على الصخور الكربونية. في هذا السياق، تم استخدام أجهزة قياس التوتر السطحي، وزاوية الاتصال، والجهد الزتّي. وقد لوحظ أن SnO_2 وTiO_2 بتركيزات (١٠٠٠ جزء في المليون) قللت التوتر السطحي بشكل كبير إلى (٣٤,٦٩، ٤٣,٤٤ ملي نيوتن/متر) مع استقرار يصل إلى (٥١,٢٧، ٤٩,١٧ ملي فولت) على التوالي. كلا النتيجتين تتجاوزان العتبة المقبولة عمومًا بحوالي ±٣٠ ملي فولت، مما يشير إلى وجود تنافر كهرستاتيكي قوي يمنع التكتل ويضمن استقرارًا طويل الأمد في التعليق. الانخفاض الكبير في زاوية الاتصال، من ٩٥,١٧° إلى ١٦,٣٢° لـ SnO_2 ومن ٩٨,٤٨° إلى ٢٥,٢٦° لـ TiO_2، يشير إلى تحول من حالة غير مبللة إلى حالة مبللة أكثر. هذا التغيير مهم لأنه يعكس زيادة في الألفة بين محلول الحمض والأسطح الصخرية الكربونية، مما يعزز توزيع الحمض بشكل أفضل ويعزز إذابة الصخور. يمكن الاستنتاج أن SnO_2 وTiO_2 قابلان للاستخدام كمواد خافضة للتوتر السطحي ذات أداء ممتاز في عملية التنشيط.

الكلمات الدالة: الموائع النانوية الحمضية، الخواص السطحية، الجهد الزتّي، زاوية الاتصال، الجسيمات النانوية لـSnO_2، الجسيمات النانوية لـTiO_2.