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To cite this article: Yaser Ahmadi, Mohsen Mansouri & Peyman Pourafshary (2024) Application of ZnO–TiO₂/zeolite nanocomposites to enhance oil recovery from sandstone formations, Petroleum Science and Technology, 42:25, 4932-4945, DOI: [10.1080/10916466.2023.2246491](https://doi.org/10.1080/10916466.2023.2246491)

To link to this article: <https://doi.org/10.1080/10916466.2023.2246491>



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Application of ZnO–TiO₂/zeolite nanocomposites to enhance oil recovery from sandstone formations

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ABSTRACT

Developing novel nanocomposites is a successful approach to affecting the nanoparticle, fluid, and rock interactions in porous media. This study introduces new ZnO–TiO₂/ZSM-5 nanocomposites (ZTZ), and the improvements compared to the standalone application of ZSM-5 zeolite, are investigated. Several methods, such as XRD, SEM, EDX, and BET, were used to study the morphology of the developed nanocomposites. Zeta potential data showed that ZTZ had better stability conditions compared to ZSM-5. The effect of ZTZ on fluid/rock interactions such as IFT, wettability, and surface charges was measured and analyzed to select the most effective nanocomposites concentration to detach oil from the surface at the reservoir conditions (1800 Psi, 60 °C). In the presence of ZTZ, wettability alteration to the wet state was greater, and IFT was reduced to lower values. Hence, ZTZ altered the capillary force more than standalone nanoparticles. This effect was also observed in oil displacement tests, where at the optimum concentration, accelerated production and a 9% higher recovery by ZTZ were observed compared to the output by ZSM-5 flooding. It was found that the ZTZ presented in this study is an efficient nanocomposite for EOR application in sandstone formations.

KEYWORDS

EOR; nanocomposites; sandstone; wettability alteration; ZnO–TiO₂/ZSM-5

1. Introduction

Nanoparticles are used to enhance oil recovery as a new chemical method. Due to the small size of nanoparticles (between 10 and 100 nm), high surface-to-volume value, and high surface energy, nanoparticles effectively change the rock/fluid interface properties. Parameters such as wettability and oil/brine IFT are considerably affected by the presence of nanoparticles (Nazarahari et al. 2020; Jafarbeigi et al. 2022; Mansouri, Ahmadi, and Jafarbeigi 2022; Ali 2023; Jafarbeigi et al. 2023; Maleki et al. 2023). The main mechanisms known in the presence of nanocomposites are disjoining

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 Supplemental data for this article can be accessed online at <https://doi.org/10.1080/10916466.2023.2246491>.

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pressure, inhibition of asphaltene, IFT, and wettability alterations (Khaksar Manshad et al. 2022). Different researchers have used nanoparticles such as TiO_2 , Zeolite, and ZnO for EOR applications. TiO_2 nanoparticles are cost-effective and stable materials for industrial applications (Kumar et al. 2020a, 2020b]. Titanium components are essential for EOR, especially to change wettability from oil wet (OW) to water wet (WW) and prevent asphaltene adsorption in porous media (Esmaeilzadeh, Sadeghi, and Bahramian 2018). Khalilnezhad et al. (2019) used TiO_2 nanoparticles to alter interfacial tension at different salinities, pressures, and temperatures. They observed that at low nanoparticle concentrations, IFT decreased with increasing concentration, while at higher values, the trend was the opposite. Moreover, it was seen that adding TiO_2 below an optimum concentration (OP) was effective, and above an optimum concentration, nanoparticles are agglomerated, and IFT is increased (Bennetzen and Mogensen 2014; Jiang, Li, and Horne 2017). Ehtesabi, Ahadian, and Taghikhani (2015) observed an increase in sweep efficiency from 41 to 55% with 0.01 wt.% TiO_2 nanoparticles. Changing wettability from OW to WW was also suggested as a mechanism for increasing sweep efficiency in the presence of TiO_2 nanoparticles. Keykhosravi, Vanani, and Aghayari (2021) used TiO_2 nanoparticles with xanthan gum for EOR in carbonate sheets. It was proven that TiO_2 altered wettability (OW to WW), and RF increased from 12 to 19%. Kumar and Sharma (2018) used TiO_2 (0.05–0.1 wt.%) to improve Silica stability in polyacrylamide. It was found that, dispersion stability was increased in the presence of the obtained solution (Kumar and Sharma 2018).

ZnO nanoparticles with small sizes between 10 and 100 nm have also been used for chemical EOR and reported in several publications (Kong and Ohadi 2010). Their biocompatibility and low toxicity make zinc oxide nanoparticles attractive for the industry (Khazaei and Hosseini 2017; Vabbina et al. 2017). It was observed that ZnO nanoparticles have an influential role in wettability alteration and reduction in oil viscosity (Sayyadnejad, Ghaffarian, and Saeidi 2008; Latiff et al. 2011; Tajmiri et al. 2015; Lee et al. 2016; Tajmiri and Ehsani 2017). ZnO 's effects on IFT are still controversial (Zaid, Latiff, and Yahya 2014; Suleimanov and Abbasov 2016). Zaid, Yahya, and Latiff (2012) used two different sizes of ZnO and Alumina nanoparticles for EOR. Based on the results, ZnO had better RF than Alumina nanoparticles.

Recently, zeolite nanoparticles have been used in the oil industry for EOR. El-Hoshoudy, Gomaa, and Taha (2019) observed an increase in recovery factor (RF) from 50.4 to 70% in the presence of zeolite at 0.01 wt.% concentration. It was observed that, with increasing zeolite nanoparticles, IFT decreased. Zhang (2014) observed higher recovery at higher

zeolite concentrations. When zeolite concentration was 0.25 wt.%, oil recovery increased from 3.8 to 29.6%. As zeolite concentration was increased to 0.5 and 1%, RFs increased from 7.9 to 33.7% and 9.1 to 34.9%, respectively.

The combination of different solid materials known as nanocomposites can be used as a novel method to enhance their performance compared to standalone applications. Effective mechanisms such as wettability alteration, capillary pressure reduction, and the development of more stable nanofluids were observed with nanocomposites. Ali et al. (2021) used $\text{TiO}_2/\text{SiO}_2/\text{Acrylamide}$ nanocomposites for EOR in carbonate oil reservoirs and observed more IFT reduction (Ali et al. 2021). Jafarbeigi et al. (2022) used ZnO-gamma alumina nanoparticles for EOR by smart water flooding.

Recently, our team worked on the development of effective nanocomposites. For example, when simultaneous water alternative associated gas (SWAG) tests were performed to improve the oil recovery, 80 ppm was selected as an OP due to the highest effect on IFT, wettability, and Z-P. At the OP and a SWAG ratio of 1:1, the tertiary RF was 27.2% at 40 °C (Ahmadi, Mansouri, and Jafarbeigi 2023a). Moreover, we used $\text{ZnO}/\text{SiO}_2/\text{Xanthan}$ as a new nanocomposites to alter wettability from OW to WW and reduce IFT at different salinities (Ahmadi, Javadi, and Kikhavandi 2023b). We recently used zeolite–zirconia–cerium oxide nanocomposites to improve the water alternative gas injection technique. Adding zirconia and cerium oxide to zeolite improved the surface area to 331.67 m²/g, and tertiary RF was 29.5% (Ahmadi and Mansouri 2021). In this study, new $\text{ZnO-TiO}_2/\text{ZSM-5}$ zeolite nanocomposites are introduced and analyzed for improving nanoparticles' characteristics, such as the zeta potential to improve oil recovery from sandstone formations at reservoir conditions. The performance of the developed nanocomposites was also compared to that of pure ZSM-5 zeolite.

2. Materials and methodology

Rock and crude oil from an Iranian sandstone reservoir have been used to perform static and dynamic tests. At a reservoir temperature of 60 °C, crude oil density, API, and viscosity are 0.846 cm/g, 28°, and 9.9 cP, respectively. The specifications of the sandstone plug used are listed in Table 1, and the oil composition properties are presented in Figure S1.

Moreover, calcium chloride, sodium chloride, magnesium chloride hexahydrate, and potassium chloride salts, purchased from the Germany Merck Company, were used to prepare the solution brines. Zeolite-titanium and zeolite-zinc oxide were synthesized based on methods described in Chang et al. (2015) and Bouvy et al. (2006), respectively. A Philips PW 1800 XRD machine recorded the X-ray diffraction pattern. FE-SEM TESCAN MIRA3

Table 1. Specifications of sandstone plug.

Properties	Measured values
Porosity (%)	21.50
Permeability (mD)	26.45
Permeability (g)	124.00
Core length (cm)	4.80
Core diameter (cm)	3.80
Oil saturated core mass (g)	134.00

performed X-ray energy diffraction analysis (EDX). The size of the holes was determined by the BET model NOVA 2000 Quantachrome. Morphological examination of products and estimation of particle size were conducted using scanning electron microscope (SEM) model Leo 1455 VP and the FE-SEM TESCAN MIRA3 device. Zeta potential (Z-P) was measured using the Malvern Zetasizer Nano ZS ZEN3600.

2.1 Synthesis of ZTZ nanocomposites

The zeolite ZSM-5 used in this work was synthesized by the method explained in Karimi et al. (2012); sol-gel was used for preparing ZTZ. To make the nanocomposites, a 1:2 weight ratio mixture of ZSM-5 and zinc nitrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, Merck, 98%) was mixed in ethanol ($\text{C}_2\text{H}_6\text{O}$, Merck, $\geq 99\%$), then titanium (IV) butoxide ($\text{C}_{16}\text{H}_{36}\text{O}_4\text{Ti}$, TBT, Sigma-Aldrich, 97%) was added to the solution in the same ratio. 1 M dilute nitric acid (HNO_3 , Merck, 65%) was added while stirring for 50 min at 30°C . The stirring continued for 2 h to prepare the gel. During the formation of the gel, the pH of the solution increased from 1 to 3. The mixture was dried at 100°C for 12 h. The obtained powder was calcined at 600°C by an electric furnace for 6 h, and ZTZ nanocomposites (25 wt.% ZnO – 25 wt.% TiO_2 –50 wt. % ZSM-5) were obtained.

2.2 Contact angle and interfacial tension measurements

Contact angle and interfacial tension were obtained with the HPHT PetroAzma model setup, as shown in Figure S2. There are two different pumps for oil and water/nanofluid; the primary main cell is filled with water/nanofluid. The liquid was released from the upper part of the cell to remove air trapped in the cells. The water pump and heating jacket set the cell at reservoir pressure and temperature, respectively. The oil droplet shape in the surrounding brine was analyzed to measure the IFT. The procedure to obtain the contact angle was the same, and the oil droplet was released into the sandstone sheets. The contact angle of the droplet on the sheets in the presence of water or nanofluid was measured. Each experiment was repeated three times, and the average results were reported. For

performing contact angles, sandstone sheets were prepared, aged at 80 °C for 7 days, rinsed with kerosene, and dried at 70 °C (Nazarahari et al. 2020). Then, for obtaining contact angle, a sandstone sheet was put in the main cell, and after applying reservoir pressure and temperature, an oil droplet was captured to obtain contact angle. For performing IFT and CA, different concentrations, including 10, 20, 30, 40, 50, and 60 ppm, were prepared by dispersing nanocomposites in brine solutions. Then, IFT and CA were obtained at 60 °C and 1800 Psi, and after matching results with Zeta potential tests, OP for nanofluids was obtained.

2.3 Nanofluid flooding

A core flooding apparatus was used to inject the nanofluid, PetroAzma model, as illustrated in Figure S3. The test device includes three transfer vessels (brine, crude oil, and nanofluid), two pumps, a core holder, an oven, pressure gauges, and a hand pump.

The overburden pressure of 2000 psi was applied, and the temperature was maintained at 60 °C in the oven. At the first step, sandstone core samples were weighted, and plugs were saturated with brine. Saturated sandstone plugs were weighted. Then, absolute permeability was obtained with Darcy's law. Next, SWir were measured by injecting oil at 1800 psi, and 60 °C. Plugs were aged at reservoir conditions of 80 °C for 7 days as CA tests, and RFs were calculated in this step as base. As it was mentioned, OPs were obtained based on previous tests, including Zeta potential and CA, which were 40 ppm. To see the effects of nanocomposites on incremental oil recovery, experiments as "a" to "h" were repeated at the same conditions, including 1800 psi, 60 °C, and the same overburden pressure of 2000 psi. Moreover, differential pressure data was obtained between the inlet and outlet of sandstone plugs.

3. Results and discussion

3.1 Nanocomposites characterizations

ZTZ crystal structures were determined with an XRD test. The XRD patterns of ZSM and the nanocomposites are shown in Figure 1. The differences between the two patterns are due to ZnO and TiO₂. The sharp peaks in the range of diffraction lines $2\theta = 5^{\circ}$ – 35° indicate the well-crystallized structure of the zeolite. The XRD of the sample synthesized in this research agrees with a similar measurement by Zhang et al. (2003). In the XRD pattern of the ZTZ (Figure 1b), the characteristic peaks of the ZnO phase are 84.31, 48.34, 62.56, 28.36, and 68.04 (Mansouri et al. 2017; Nodehi, Atashi, and Mansouri 2019). The characteristic peaks of the TiO₂ phase are located

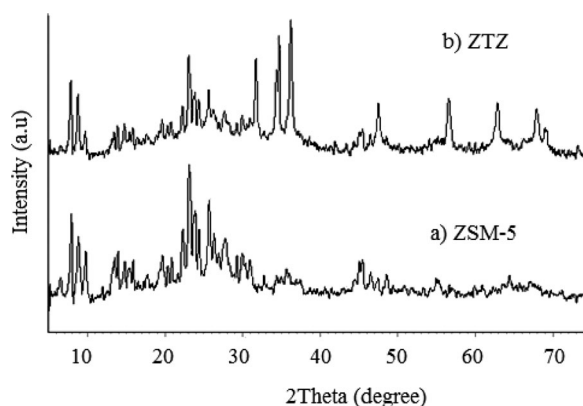


Figure 1. XRD patterns of (a) ZSM-5 zeolite and (b) ZTZ.

at 37.25° , 47.23° , 54.69° , 62.25° , and 72.93° (Sakthivel et al. 2003), which is proof of the synthesis. The average crystal size of nanocomposites was calculated using Scherer's formula, 19.13 nm.

The specific surface area and average pore diameter of synthesized zeolite by BET test were equal to $369.38 \text{ m}^2/\text{g}$ and 1.04 nm, respectively, which indicate the porous structure of zeolites. After fixing zinc oxide and titanium nanoparticles on zeolite and calcining it, its specific area decreased to $322.34 \text{ m}^2/\text{g}$ as parts of the zeolite surface were covered. The average diameter of the pores of ZTZ was 1.22 nm, which shows the nano-porosity of the material. The morphology of zeolite and ZTZ was investigated by an electron microscope (FE-SEM, SEM) (Figure 2). The zeolite sample is composed of a large number of cubic units attached. The uneven surfaces of the substrate and pores, which can be seen, are reasons for the high specific surface area and absorption capacity. As shown in Figure 2b, zinc oxide and titanium nanoparticles are placed on the surfaces and pores of the zeolite substrate and have dimensions of less than 40 nm.

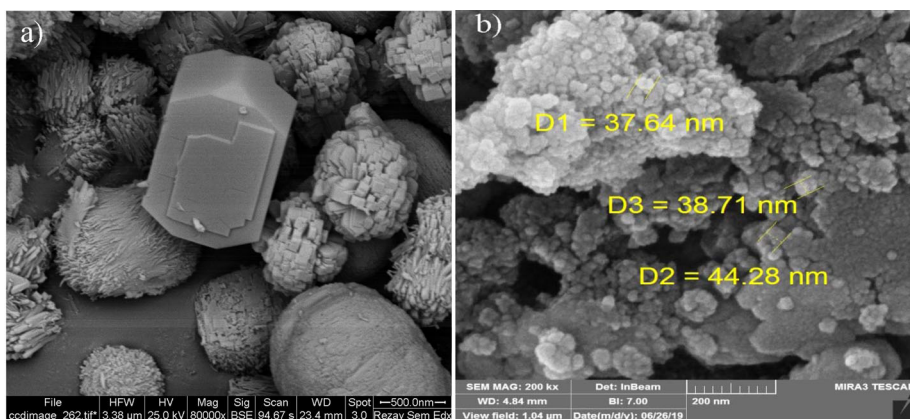


Figure 2. (a) SEM image of ZSM-5 zeolite and (b) FE-SEM image of ZTZ.

The chemical composition of the ZSM-5 zeolite and ZTZ nanocomposites was investigated with the X-ray energy diffraction (EDX) technique. According to the results of EDX in Figure 3, elements Ti, Zn, and O with weight percentages of 12.81, 11.92, and 41.52 were present on the ZTZ nanocomposites, respectively.

3.2 Nanofluids specifications

The oil/nanofluid/rock properties of ZTZ and ZSM-5 were measured and compared by different parameters such as IFT, CA, density, pH, Z-P, and viscosity at 10–60 ppm ZTZ and ZSM-5 and 1800 psi and 60 °C, as shown in Table 2.

Generally, as ZTZ and ZSM-5 concentrations increased from 10 to 60 ppm, the viscosity and density of the nanofluid slightly increased. Z-P is an indicator of nanofluid stability. Figure 4a shows the Z-P values of ZTZ and ZSM-5 nanofluids. For both cases, Z-P was less than -30 , which shows a stable fluid (Hendraningrat and Torsæter 2015; Li et al. 2018; Ali et al. 2019). Z-P data showed that ZTZ had better stability conditions than ZSM-5, especially at concentrations of about 40 ppm.

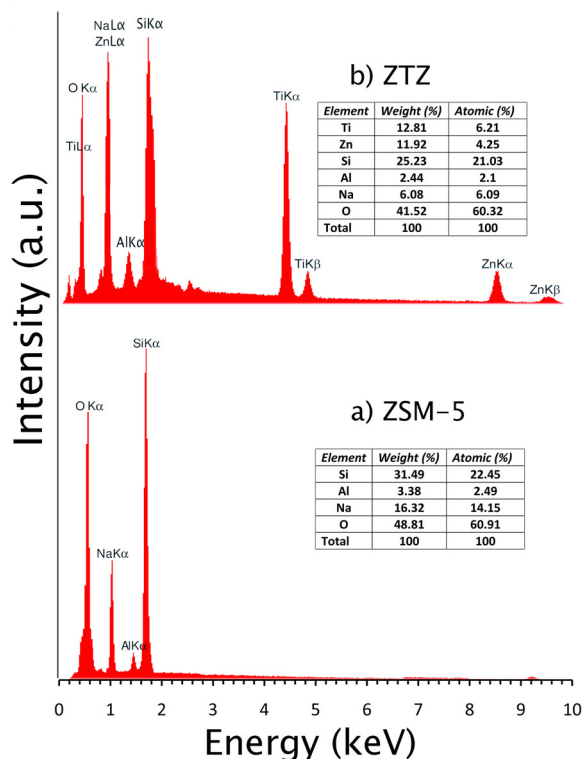
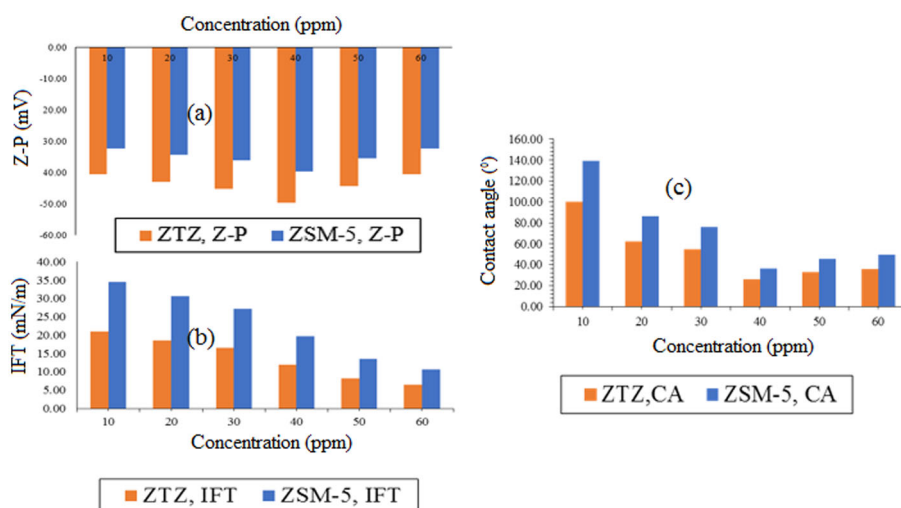


Figure 3. X-ray energy diffraction analysis of (a) ZSM-5 zeolite and (b) ZTZ.

Table 2. Properties of the nanofluid prepared with different concentrations.

	N-particles concentration (ppm)	Interfacial tension (mN m ⁻¹)	pH (–)	Density (g/cc)	Viscosity (cP)	Z-P (mV)	CA ^a (°)
ZTZ	10	20.95	7.50	0.9992	2.20	–40.44	100.30
	20	18.58	7.36	0.9993	2.23	–42.91	61.91
	30	16.48	7.03	0.9995	2.27	–45.15	54.71
	40	11.91	6.69	0.9996	2.28	–49.68	26.23
	50	8.19	6.21	0.9997	2.30	–44.24	32.86
	60	6.48	5.95	0.9999	2.32	–40.44	35.46
ZSM-5	10	34.57	8.55	0.9991	1.91	–32.35	139.41
	20	30.65	8.39	0.9992	1.94	–34.33	86.05
	30	27.19	8.01	0.9993	1.97	–36.12	76.05
	40	19.65	7.63	0.9994	1.98	–39.75	36.46
	50	13.51	7.08	0.9995	2.00	–35.39	45.68
	60	10.69	6.78	0.9996	2.02	–32.35	49.29

^aOriginal CA without nanoparticles:150.35°.


Figure 4. (a) Zeta potential, (b) IFT, and (c) CA tests in the presence of ZTZ and ZSM-5.

A layer of nanocomposites on sandstone plugs has effects on IFT and contact angle, which are dominant capillary parameters that control the residual oil in the EOR. The alteration of these parameters depends on the type and properties of nanoparticles, concentration, and rock/fluid surface charges (Khazaei and Hosseini 2017; Vabbina et al. 2017). Figures 4b and c show contact angle and IFT with ZTZ and ZSM-5. ZTZ decreased IFT and altered CA more than ZSM-5. For example, at 40 ppm, ZTZ changes the IFT to 11.91 mN/m, which is about 40% lower than the IFT by ZSM-5. The same point was observed by CA measurement, which shows the most significant change at 40 ppm, which makes the rock surface water wet and was OP in this study.

The OP was also confirmed by the stability criteria. Figure 5 shows the contact angle and Z-P values on the same graph. It can be seen that at 40 ppm, the highest stability and wettability alterations occurred.

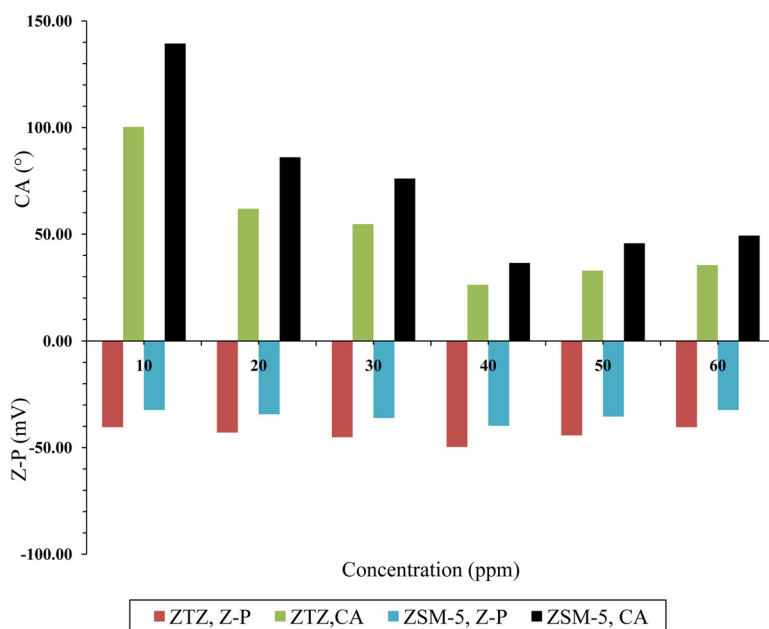


Figure 5. Selecting an optimum concentration for nanofluid.

3.3. Displacement tests

The designed nanofluids were used at OP for oil displacement tests. Figures 6a and b depict how nanocomposites affected RF and differential pressure at 40 ppm for ZTZ and were compared to ZSM-5 nanofluid as a base. The RF with ZTZ was 60.90%, higher than the 50.51% achieved by ZSM-5 at 60 °C and 1800 psi. Moreover, differential pressure results for ZTZ were lower than ZSM-5.

Two mechanisms were observed to enhance the oil recovery. The pressure drop developed by nanocomposites was much higher early on resulting in more viscous force and higher recovery. Wettability alteration was another dominant mechanism, which was stronger in nanocomposites than pure ZSM-5 zeolite, as proved by CA results. The effect of wettability alteration in adsorbing oil with nanoparticles, separating nanocomposites, and adsorbing on the rock surface is schematically shown in Figures 7a, and b, respectively. In this study, three main parameters were known to be effective in increasing incremental oil recovery: IFT reduction, wettability alteration, and zeta potential tests. We found that Z-P and contact angle were effective parameters for selecting OP. In other words, wettability alteration from OW to WW and stable conditions of nanoparticles in solution are dominant effective parameters. By the end, we think that selecting an OP can be an effective method for increasing RF in sandstone porous media in the presence of proposed nanocomposites.

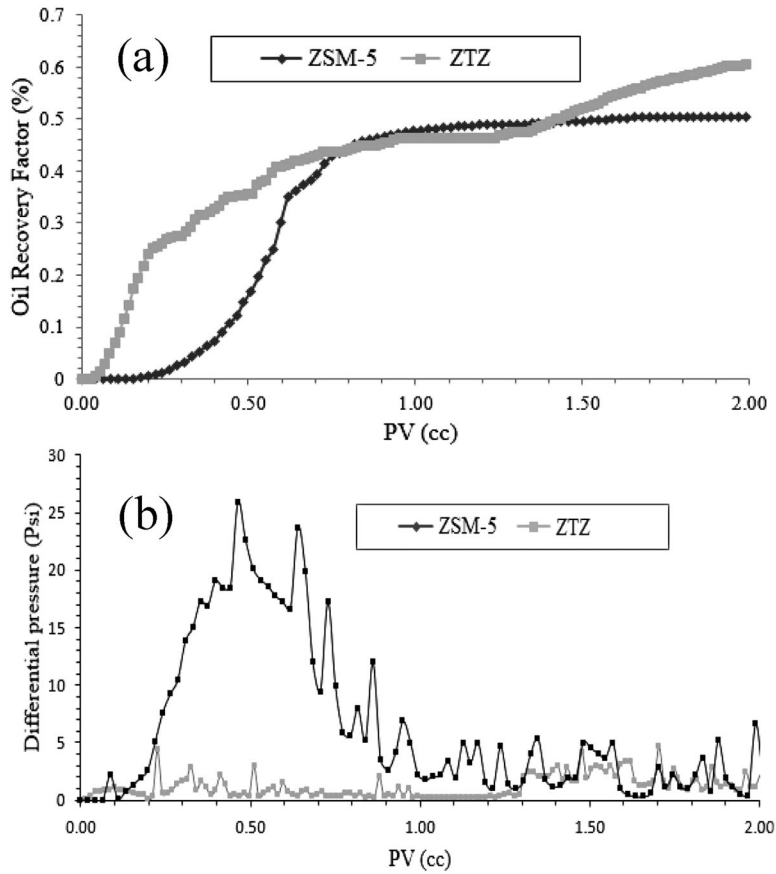


Figure 6. (a) Recovery factor and (b) comparison differential pressure during ZTZ and ZSM-5 nanofluids flooding.

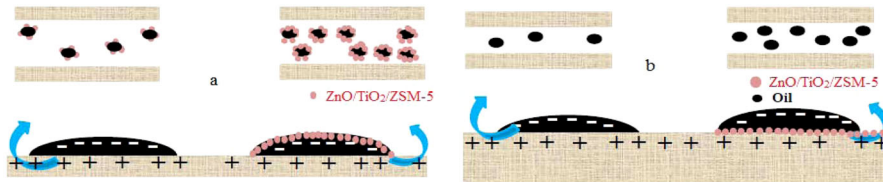


Figure 7. Oil recovery mechanisms.

4. Conclusion

In this paper, ZTZ and ZSM-5 were characterized by XRD, EDX, BET, and SEM. Then various IFT, contact angle, viscosity, and Z-P tests were obtained using ZTZ and ZSM-5 at concentrations of 10, 20, 30, 40, 50, and 60 ppm at 1800 Psi and 60 °C. Below are the main results obtained:

- ZTZ had a lower specific surface area and a higher pore diameter when compared to ZSM-5

- CA and Z-P were found to be the two main factors for detecting an OP in flooding tests.
- 40 ppm provides the best values, and it has the highest stability and wettability alteration for both ZTZ and ZSM-5.
- At 40 ppm, surface charge values of -49.68 and -39.75 were achieved for ZTZ and ZSM-5 by Z-P measurement, proving the high stability of both nanoparticles, proving the high stability of both nanoparticles and the higher stability of ZTZ compared to ZSM-5.
- At OP = 40 ppm, CA values show the highest changes, and they were 26.23° and 36.46° for ZTZ and ZSM-5, respectively.
- At OP = 40 ppm, the RF increased from 50.51 to 60.90% in the presence of ZTZ and ZSM-5, respectively at 60°C and 1800 psi.
- During core flooding tests, it was confirmed that, CA had the main reason for having higher oil recovery, seen during ZTZ comparison to ZSM-5 at an OP = 40 ppm and reservoir conditions.
- Synthetic ZTZ was known as an effective nanocomposites in core flooding in sandstone porous medium.

Author contributions

YA: data curation, writing—original draft, visualization, investigation. MM: conceptualization, methodology, software, supervision, writing—review and editing. PP: writing—review and editing.

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Nomenclature

BET	Brunauer–Emmett–Teller
CA	Contact angle
EDX	Energy-dispersive X-ray spectroscopy
EOR	Enhance oil recovery
IFT	Interfacial tension
OP	Optimum concentration
SEM	Scanning electron microscope
SWAG	Simultaneous water alternative associate gas
TBT	Tetrabutoxytitanium
XRD	X-ray powder diffraction
Z-P	Zeta potential
ZTZ	ZnO–TiO ₂ /ZSM-5 nanocomposites