Synthesis of Zinc Oxide Nanoparticles for Oil Upgrading and Wax Deposition Control: Effect of Calcination Temperature

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Abstract: *In this study, ZnO nanoparticles were synthesized using a sol-gel method for oil upgrading and wax deposition control. The synthesized ZnO nanoparticles were used to measure viscosity and wax deposition in the heavy crude oil and to investigate the effectiveness of the nanoparticles in the reduction of viscosity and wax deposition control of the heavy crude oil. This study investigated the effect of calcination temperature on ZnO nanoparticles during synthesis towards viscosity reduction and wax deposition control. ZnO nanoparticles were calcined at different temperatures ranging from 300 to 900 °C. The calcined ZnO nanoparticles were characterized using X-ray diffraction (XRD), Field Emission Scanning Electron microscope (FESEM), and Energy-dispersive X-ray spectroscopy (EDX) for its structure, size, shape, and morphology. The characterization results showed a hexagonal wurtzite structure of ZnO nanoparticles. The physical properties and rheology of heavy crude oil were characterized by using Electronic Rheometer and cold finger method to analyze the viscosity, shear rate, and wax deposition of the heavy crude oil for performance study. Decreased in crystallite size from 15.59 to 12.84 nm was observed with increasing calcination temperature from 300 to 400 °C, and a further increase of calcination temperature from 400 to 900 °C, the crystallite size increased from 12.84 to 41.58 nm. The degree viscosity reduction (DVR %) of heavy crude oil was observed to increase by 41.7%, with decreasing ZnO nanoparticles size from 30.11 nm to 12.84 nm. The optimum calcination temperature was 400 °C. Wax deposition decreases by 32.40% after the addition of ZnO nanoparticles into heavy crude oil.*

Keywords: *zinc oxide (ZnO); nanoparticles; calcination; wax content; viscosity reduction*

■ INTRODUCTION

Wax deposition in heavy crude oil has always brought a significant impact on the petroleum industry, especially in the transportation and production section. There are various effects of wax deposition in heavy crude oil that we should be concerned about, for example, non-Newtonian flow characteristics of the fluid, increased pumping power, decreased flow rate, or even to the total blockage of the pipeline [1]. Wax precipitation induced by the viscosity of heavy crude oil that increases can cause flow line pressure to drop, leading to low flow rates [2]. In turn, this makes conditions for wax deposition in the pipes more favorable. Serious wax deposition in heavy crude oil needs extra attention since it may cause harm not only to the transportation and production section, but also to the petroleum economy as well. There are many advanced technology and chemicals for wax prevention and removals, such as wax crystal modifiers and dispersants. However, these methods have disadvantages, such as cost and limited to one well [1]. ZnO nanotechnology has introduced a more effective solution for the petroleum industry as it provided simple and cheaper technology. Hence, it has become a major interest nowadays to reduce wax deposition from the heavy crude oil, to avoid its bad effect on the petroleum industry as well as to the environment.

Wax deposition in pipelines is commonly caused by several factors such as temperature, flow rate, oil

composition, and shear rate [3]. Generally, wax precipitation and deposition are related to the wax content of heavy crude oil. As the temperature increases, the viscosity of the crude oil decreases. In oil composition, the API gravity plays an important role in the petroleum industry as it is related to the wax deposition [4]. A high API gravity shows that crude oil has low wax content. An API gravity that is greater than 10 indicates light crude oil, whereas an API gravity of less than 10 indicates heavy crude oil [5]. Basically, the heavier the crude oil, the higher wax content.

In this study, the focus will be given on the heavy crude oil from the petroleum industry, such as in transportation and production. It is well known that this heavy crude oil releases a higher significant deposition of wax content. The wax that is present in the heavy crude oil primarily consists of paraffin hydrocarbons (C_{18}–C_{36}) known as paraffin wax and naphthenic hydrocarbons (C_{30}–C_{60}) [6]. The factor that leads to wax deposition problems depends on the type of oil and the molecular composition of the wax molecules. The waxes in crude oils are often more challenging to control because the alkane chains are often longer in the crude oil [7].

Currently, various conventional methods have been used to remove the wax precipitate in heavy crude oil, such as thermal techniques, chemical techniques, mechanical techniques, and thermo-chemical packages [1]. However, these conventional methods have some downside with their process in removing or preventing the wax precipitate from heavy crude oil [8]. For example, electric heaters can cause an increase in maintenance costs, and the availability of electrical power is limited, while chemical techniques, such as dispersants, could lead to harmful erosion [1].

Numerous transition metals have been used in many applications such as iron and zinc because of its heat and electric conductivity [9]. In addition, these metals are commonly used as catalytic agents due to their ability to change their state or absorb other substances on their surface and activate them in the process [9].

Numerous methods have been described in research to synthesize ZnO nanoparticles, such as the sol-gel method, co-precipitation, and spray pyrolysis [10]. However, in this study, the sol-gel process was more preferred than the other methods because of its low cost and simple [11].

■ EXPERIMENTAL SECTION

Materials

Crude oil obtained from Kemaman Bitumen Company Sdn. Bhd. (KBC) was used as received for characterization and rheological tests to analyze its properties. Zinc acetate dihydrate ($Zn(CH_3COO)_2{\cdot}2H_2O$), oxalic acid ($C_2H_4O_4{\cdot}2H_2O$), and ethanol ($C_2H_5OH$) 95% AR Grade obtained from Vchem Laboratory Chemicals, ammonia solution acquired from Daejung Chemicals. The other chemicals such as hydrochloric acid (HCl), methanol (CH_3OH), *n*-heptane (C_7H_{16}), and toluene (C_7H_8) also obtained from the Vchem Laboratory Chemicals.

Instrumentation

High-Resolution X-ray Diffractometer (PANalytical X'Pert PRO MRD) was used to analyze the crystallinity of the zinc oxide nanoparticles. Energy Dispersive X-ray Spectroscopy (EDX) was used in this study to verify the elemental composition of nanoparticles sample. The surface morphology of ZnO nanoparticles was determined using Field Emission scanning electron microscopy (FESEM). The rheological measurement was carried out using a Rheometer Paar Physica MCR300.

Procedure

Characterization of crude oil

The sample of crude oil studied in this work was obtained from Kemaman Bitumen Company (KBC) Sdn Bhd. Table 1 shows the physical properties and composition of crude oil in weight percentage (%) of the sample crude oil that was obtained from KBC. The density of crude oil was determined by using Eq. (1) and Eq. (2) where S.G., ρ_{oil}, and ρ_{water} are the specific gravity, oil density, and water density, respectively. The API gravity of crude oil refers to the density of the fluid after the gas been liberated from the fluid at ambient pressure and reservoir temperature [12]. Crude oil gained from the KBC was considered heavy crude oil because of API

Table 1. Physical properties and composition of crude oil

Physical properties	
Density (kg/m^3)	999
Specific gravity, SG	0.999
API°	11
Viscosity at 40 °C (cP)	17,751
Color	Black
Cloud Point (°C)	12
Composition	Wt.%
Saturates	3.0
Aromatic	63.4
Resin	12.9
Asphaltenes	20.7

and density value of mostly heavy oil ranging between 10° to 22° API and 920 to 1000 kg/m^3 [13].

$$API° = \frac{141.5}{S.G} - 131.5 \quad (1)$$

$$S.G. = \frac{\rho_{oil}}{\rho_{H_2O}} \quad (2)$$

Synthesis of zinc oxide nanoparticles

ZnO nanoparticles were prepared under constant processing conditions of pH, zinc acetate, oxalic acid ratio, and drying temperature by using a sol-gel method. Zinc acetate [Zn $(CH_3COOH)_2{\cdot}2H_2O$], oxalic acid [$C_2H_2O_4$], and ethanol [CH_3CH_2OH] were used as precursor materials for the preparation of ZnO nanoparticles [10,14]. In addition, the effect of calcination temperature was studied during the synthesis of ZnO nanoparticles by varying its temperature from 300 to 900 °C [15-16]. For the preparation of ZnO nanoparticles with molar ratios of 1:2 of zinc acetate and oxalic acid, 2 g of zinc acetate was added to the 100 mL of ethanol solution (+ 10% volume of water) in 500 mL glass beaker in a water bath at 65 °C under reflux condition for 30 min. An amount of 1.64 g of oxalic acid powder was added to the 100 mL of ethanol solution in 500 mL glass beaker at 45 °C under 700 rpm speed for 30 min. Then, the oxalic acid solution was slowly added by using a burette to the zinc acetate solution in 500 mL glass beaker under vigorous stirring at 1000 rpm [10,17]. The final pH of the reactant was kept at 3 by adding a required amount of hydrochloric acid and ammonia solution, respectively. Then, the solution was kept undisturbed for a while till white precipitates were seen in the solution and filter the precipitate by using a vacuum pump. The precipitate was dried at 80 °C for 2 h in a sintering boat which was put in a drying oven, and then calcined at 300 °C for 2 h in a sintering boat in the furnace to obtain a smoother powder [11]. The calcination of the ZnO precipitate was repeated but at different calcination temperatures between 400 to 900 °C.

Characterization of ZnO nanoparticles

The X-ray diffraction analysis was carried out for the synthesized ZnO nanoparticles by using a High-Resolution X-ray Diffractometer (PANalytical X'Pert PRO MRD) with Cu Kα radiation (λ = 1.54060 Å) over the angle 2θ range of 10°–90° [10]. X-ray diffraction was used to analyze the crystallinity of zinc oxide nanoparticles. The XRD patterns indicate the formation of the crystal structure. The diffraction angle used for the ZnO nanoparticles sample was from 10° to 90°. In this study, the intensity peaks and diffraction angle of calcined ZnO at 300 to 900 °C were investigated to determine the effect of calcination temperature on the crystallite size of ZnO nanoparticles.

Other than XRD, field-emission scanning electron microscope (FESEM) and energy dispersive X-ray (EDX) were also used to study the characteristic of these nanoparticles by analyzing their morphological structure and composition element of the ZnO nanoparticles. The morphological structure of ZnO nanoparticles was determined by using a high-resolution scanning electron (Zeiss, Supra40VP) with the magnification of 10,000.

The crystallite size of ZnO nanoparticles was then estimated by using the Debye-Scherer formula given in Eq. (3), where 0.89 is Scherer's constant, λ is the wavelength of X-rays, θ is the Bragg diffraction angle, and B is the full width at half-maximum (FWHM) of the diffraction peak [18].

$$D = \frac{0.89\lambda}{B\cos\theta} \quad (3)$$

Rheological measurements

ZnO nanoparticles (0.4 g) was added to the heavy crude oil. The tests were performed at a different temperature ranging between 30, 45, and 60 °C. This

experiment was run in a controlled condition: shear rate between 0 and 500 s^{-1} at 24.85 °C. Shear rate and apparent viscosity values were obtained every 10 sec, resulting in 28 points, respectively. For performance study, the effect of ZnO nanoparticle's size and temperature at a higher shear rate on the viscosity reduction of heavy crude oil were investigated by using this equipment.

■ RESULTS AND DISCUSSION

Characterization of Synthesized Zinc Oxide Nanoparticles

X-ray diffraction (XRD)

XRD was used to characterize the crystalline nature of the ZnO nanoparticles. Fig. 1 shows XRD patterns of ZnO nanoparticles calcined at different temperatures ranging from 300 to 900 °C. The presence of high-intensity peaks corresponds to the (100), (002), and (101) lattice plane confirms the formation of the hexagonal wurtzite structure of the ZnO nanoparticles. The peaks gained in this study perfectly matches with the standard peaks of ZnO powder diffraction (JCPDS 36-1451). All the diffraction peaks of ZnO nanoparticles at different calcination temperatures were observed at 2θ: 31.95°,34.7°, 36.4°, 47.8°, 56.9°, 63.1°, 66.5°, 68.1°, and 69.3°, that attributed to the (100), (002), (101), (102), (110), (103), (200), (112), and (201) crystal planes of the hexagonal ZnO powder. Fig. 1 suggests that, with an increase in calcination temperature, the intensity of diffraction peaks increases, which indicates the strengthening of the ZnO phase [11].

The obtained crystallite size of ZnO nanoparticles was tabulated in Table 2. The decrease in crystallite size from 15.59 to 12.84 nm was observed with an increase in calcination temperature from 300 to 400 °C. However, the crystallite size continued to increase from 12.84 to 41.58 nm by a further increase in calcination temperature from 400 to 800 °C. The average crystallite size of ZnO nanoparticles was found to be 25.42 nm. The decrease in crystallite size at low temperature indicates the restructuring process, whereas the increase in calcination temperature with an increase in crystallite size suggests the strengthening of the ZnO nanoparticles phase [11]. In addition, by referring to Fig. 1, the spectrum became sharper, and diffraction peak became narrower with the increase in the temperature from 400 to 800 °C, indicating that the crystallite ZnO nanoparticles formation has been established due to growth rate between the crystallographic planes [16,19-21].

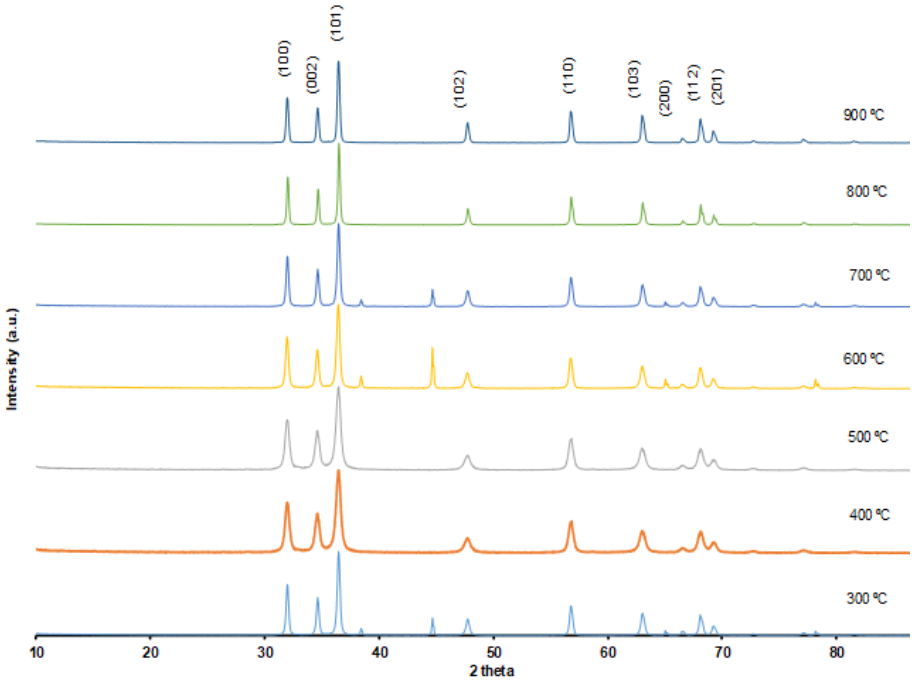

Fig 1. XRD patterns of the ZnO nanoparticles calcined at 300, 400, 500, 600, 700, 800, and 900 °C.

Table 2. XRD analysis of ZnO nanoparticles: Full width at half-maximum (B) and size (nm) for each degree

Temperature (°C)	Position 2θ (°)	Full width at half-maximum (B)	Size (nm)
300	36.4	0.56	15.59
400	36.4	0.68	12.84
500	36.4	0.47	18.58
600	36.4	0.34	25.68
700	36.4	0.29	30.11
800	36.4	0.21	41.58
900	36.4	0.26	33.58

Energy dispersive X-ray spectroscopy (EDX)

EDX was used in this study to verify the elemental composition of the nanoparticles synthesized by using the sol-gel method. Table 3 shows the weight and atomic percentage of EDX of the ZnO element, which suggests the good purity of the ZnO powder. Therefore, the experimental synthesis of zinc oxide nanoparticles sample was successful due to the presence of zinc and oxygen elements, respectively.

These results show that the final product was pure ZnO nanoparticles. According to Al-Hada et al. [19], by using zinc nitrate as a precursor, the atomic percentages of Zn and O were approximately 50.13% and 49.87%. From this comparison, it showed that the precursor of zinc acetate produces a higher atomic percentage of Zn than precursor zinc nitrate. However, the value of the atomic percentage of Zn and O between both precursors did not create a huge difference.

Effect of Calcination Temperature on Size of Zinc Oxide Nanoparticles

Fig. 2 shows the crystallite size of the ZnO as a function of calcination temperature ranging between 300 to 900 °C. As calcination temperature increases from 300 to 400 °C, the crystallite size of ZnO decreases. The decrease in the crystallite size of ZnO from 15.59 to 12.84 nm was due to the structural rearrangement, which indicates a restructuring process [10-11]. However, the crystallite size of ZnO tends to increase from 12.84 to 41.58 nm with increasing calcination temperature from 400 to 800 °C, which implies the strengthening of the ZnO phase. This was because of the increment of the crystallite volume to the surface ratio [11,19]. The minimum crystallite size of ZnO in this study was achieved at a temperature of 400 °C, whereas the maximum one was achieved at 800 °C.

Effect of Calcination Temperature on the Morphology of the Nanoparticles

Fig. 3 shows FESEM images of the ZnO nanoparticles at different calcination temperature. Fig. 3(a) reveals that the synthesized sample has nanoparticles appearance with a rod-like morphology structure at lower temperature. Fig. 3(b) shows that the length of the

Table 3. Weight and atomic percentage of EDX of ZnO element

Element	Wt.%	Atomic %
O	22.59	45.61
Zn	77.41	54.39

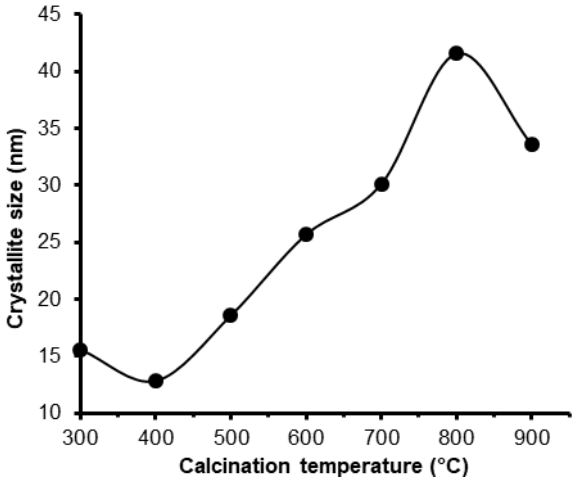

Fig 2. Variation in crystallite size with varying calcination temperatures.

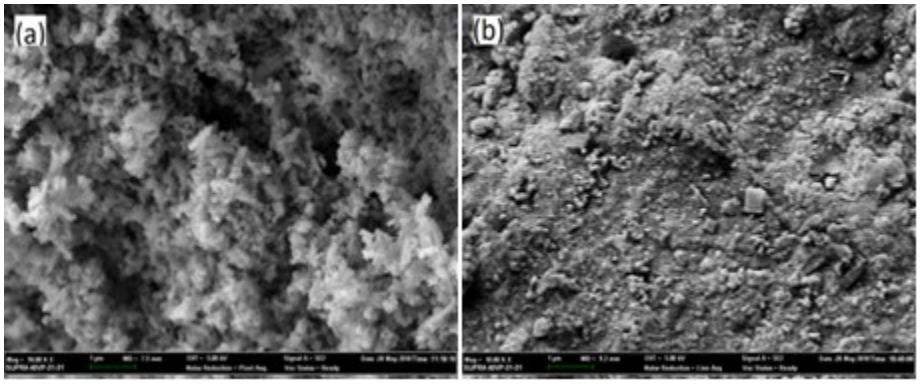

Fig 3. FESEM images of ZnO nanoparticles at calcination temperature of (a) 400 °C and (b) 500 °C.

structure was reduced with the increase in the calcination temperature. The results are consistent with previous research stated that ZnO nanoparticles product started to crumble and overlapped in a proportional relation with the increase of calcination temperature [19-20,22].

Effect of Nanoparticle Size towards Viscosity Reduction

To further investigate the effect of the calcination temperature of the ZnO nanoparticles on the heavy crude oil, the reduction of viscosity of heavy crude oil was carried out. Fig. 4 shows the viscosity of the heavy crude oil in the presence of ZnO nanoparticles at different nanoparticle sizes at 30 °C and shear rate between 0 and 80 s^{-1}. This indicates that the addition of ZnO nanoparticles into the heavy crude oil will reduce the viscosity of the heavy crude oil. The nanoparticle sizes evaluated in this study were 12.84, 18.58, 25.68, 30.11, 33.58, and 41.58 nm. It was observed that the viscosity of heavy crude oil decreased with a decrease in nanoparticle sizes. The highest viscosity reduction of heavy crude oil obtained at 12.84 nm. As particle size increases, a decrease in performance was noticed [23]. This is due to fewer active sites available for nanoparticles to attach to heavy crude oil components [24].

The degree of viscosity reduction (DVR) was calculated by Eq. (4), where μ_{HO} and μ_{np} were the crude oil before and after additional nanoparticle values, measured at a shear rate between 0 and 80 s^{-1}, respectively. Fig. 5 indicates the degree of viscosity reduction of heavy crude oil with the presence of the ZnO nanoparticles with different sizes, at 30 °C and shear

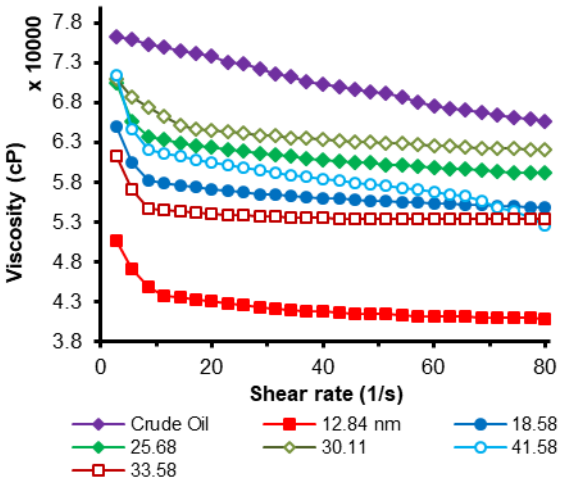

Fig 4. Viscosity of heavy crude oil in the presence of ZnO nanoparticles at different nanoparticles size at 30 °C and shear rate between 0 and 80 s^{-1}

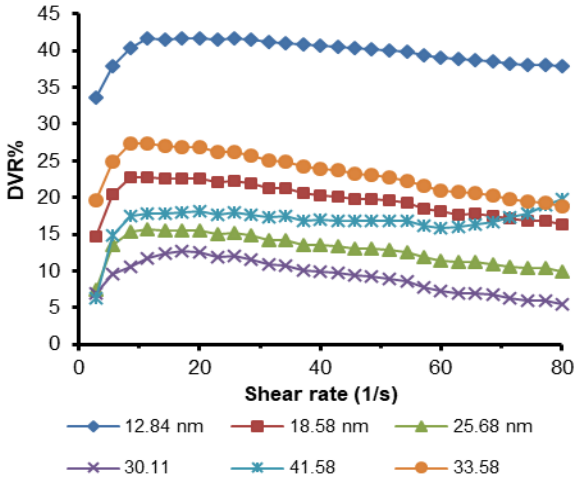

Fig 5. The degree of viscosity reduction of heavy crude oil with the presence of the ZnO nanoparticles with different sizes, at 30 °C and shear rate between 0 and 80 s^{-1}

rate between 0 and 80 s^{-1}. The values of DVR indicate that the optimal nanoparticle size at which the biggest change in viscosity obtained was 12.84 nm for all shear rates. The lowest degree of viscosity reduction was 5.5%, which occurred at 30.11 nm, while the highest degree of viscosity reduction was 41.7% at 12.84 nm. However, increasing the shear rate from 10 to 80 s^{-1} slightly reduced the DVR%.

$$\text{DVR } \% = \left(\frac{\mu_{HO} - \mu_{np}}{\mu_{np}} \right) \times 100 \quad (4)$$

Effect of Temperature and High Shear Rate on Heavy Crude Oil

Temperature plays an important role in viscosity control of heavy crude oil, as increasing the temperature will reduce the viscosity of the oil. Fig. 5 shows that ZnO nanoparticles at 12.84 nm exhibit the best performances due to the higher percentage in viscosity reduction. Therefore, in this analysis of temperature and high shear rate effect, nanoparticles with a size of 12.84 nm were chosen to be evaluated at three different temperatures at high shear rates. The temperatures of 30, 45, and 60 °C were evaluated respectively, at shear rates between 0 and 500 s^{-1}. Fig. 6 displays the rheological responses for heavy crude oil both with and without additional ZnO nanoparticles at 30, 45, and 60 °C, at shear rates between 0 and 500 s^{-1}. The DVR for 30, 45, and 60 °C, and at shear rates between 0 and 500 s^{-1} are presented.

The rheological responses of heavy crude oil with additional ZnO nanoparticles at 30, 45, and 60 °C shows the degree of viscosity reduction for heavy crude oil where the highest DVR was 71%. Therefore, increase in the shear rate from 0 to 500 s^{-1} with increase in the temperature at 30, 45, and 60 °C will decrease the DVR. The rheological behavior of heavy crude oil at 60 °C shows the minor changes in viscosity values between presence and absence of ZnO nanoparticles. Fig. 6 indicates that at temperature 30 °C, the DVR tends to increase up to 71% with increasing shear rate from 100 to 400 s^{-1}. However, at temperatures of 45 and 60 °C, the DVR tends to decrease with an increase in shear rate from 100 to 400 s^{-1}. From this observation, it can be concluded that the optimum temperature of this performance was at 45 °C, where the

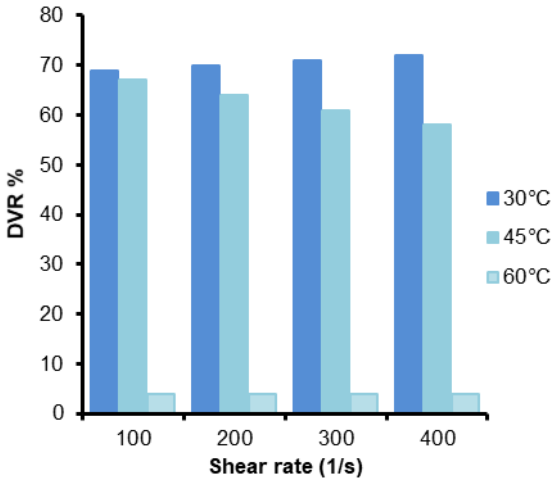

Fig 6. Degree of viscosity reduction at shear rates between 0 and 500 s^{-1} at different temperatures of 30, 45 and 60 °C

DVR was reduced nearly to 0% with further increase in the temperature. As the temperature increases, so does the rate of reaction of ZnO nanoparticles towards heavy crude oil. However, very high temperatures can denature the ZnO nanoparticles and change both the chemical and physical properties of the ZnO nanoparticles.

■ CONCLUSION

ZnO nanoparticles were successfully synthesized by using the sol-gel method at different calcination temperatures from 300 to 900 °C. The effect of calcination temperature on the structure and crystallite size of ZnO nanoparticles at different temperatures was studied by using XRD, FESEM, and EDX. The XRD results approved the presence of the formation of the hexagonal wurtzite structure of ZnO nanoparticles. The crystallinity increased with increasing calcination temperature. The crystallite size decreased (15.59 to 12.84 nm) with an increase in calcination temperature from 300 to 400 °C. Further increase in calcination temperature from 400 to 800 °C resulted in an increase in the crystallite size from 12.84 to 41.58 nm. The synthesized sample was then used for viscosity reduction for oil upgrading, and it showed that nanoparticles at the size of 12.84 nm gave the best performances for viscosity reduction compared to the others.

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