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INHIBITIVE EFFECT OF METAL OXIDE NANOPARTICLES ON WAX PRECIPITATION IN A NIGER DELTA CRUDE OIL

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Abstract

The original equilibrium conditions of crude oil in the reservoirs are always altered during transportation such that at the onset of wax appearance temperature (WAT), the solubility of wax in crude oil can no longer be sustained causing the wax to precipitate. Wax precipitation and deposition gradually block the transport facilities, thereby causing flow assurance problems. Polymeric wax inhibitors have been used and investigated; the investigation includes its synergy with nanomaterials at improving wax inhibition. But polymeric wax inhibitors have environmental issues. Hence, this work expands research scope by experimentally investigating the potentials of aluminium oxide and copper oxide nanoparticles in wax precipitation inhibition.

Density, specific gravity, API gravity, viscosity and cloud point of a waxy crude oil sample were determined. The blank crude oil sample was poured into a test jar. The test jar was closed tightly with a cork carrying a thermometer. To facilitate effective cooling, the test jar was then placed in a methanol-filled bath inside a cloud point tester. As temperature drops inside the cloud point tester, the test jar was removed gently and quickly at three minutes intervals for inspection. The cloud point (WAT) was obtained as 29.4°C. To determine the effect of temperature on wax precipitation in the blank crude oil sample, temperature points (below the WAT) outside the cloud/pour point tester considered are 20, 10, 0, -10, and -20°C. The effects of metal oxide nanoparticles concentration range of 0.1 to 1.0 g on wax precipitation in the crude oil samples at these temperatures were also analysed. The decrease in temperature was found to increase wax precipitation. Reducing temperature of blank crude oil from room temperature of 35°C to 20°C and -20°C in the cloud point tester causes wax precipitation to increase from 0 g to 0.24 g and 0.54 g respectively. For both metal oxide nanoparticles, increase in nanoparticles concentrations was found to reduce wax precipitation. At 20°C, wax precipitation reduces from 0.24 g in blank crude oil to 0.16 g and 0.20 g with 0.40 g aluminium oxide and 0.40 g copper oxide nanoparticles addition respectively. At 20, 10, 0, -10 and -20°C, the optimal aluminium oxide nanoparticles concentrations inhibiting wax precipitation are 0.4, 0.3, 0.4, 0.6 and 0.7g with corresponding minimal wax precipitations of 0.16, 0.22, 0.23, 0.31 and 0.32 g. However, at the respective temperatures, the optimal copper oxide nanoparticles concentrations inhibiting wax precipitation are 0.3, 0.4, 0.4, 0.5 and 0.6g with corresponding minimal wax precipitations of 0.20, 0.23, 0.28, 0.35 and 0.38 g. Hence, this study shows that aluminium oxide nanoparticles are more effective than copper oxide nanoparticles in reducing wax precipitation and deposition in crude oil.

Keywords: *Crude oil, Wax precipitation, Wax appearance Temperature WAT, Metal oxide nanoparticles.*

1. Introduction

Global demand for energy is ever increasing, and crude oil (and natural gas) production has really contributed to meeting this demand. Substantial numbers of developed oilfields in various regions of the world are of waxy crude oils [1-3]. Nigeria has a remarkable reserve of paraffinic crude oils with moderate to high wax content but with good quality in terms of low sulphur content and high API gravity [4]. Nigerian crude oil wax deposits were reported to be in the range of 30 to 35 % [5-8].

Often, crude oil is produced in remote locations away from where it is needed. Hence, getting crude oil to the market requires various storage and transportation networks. The original equilibrium conditions of crude oil in the reservoirs are always altered during transportation through the wellbore, the production tubing, the pipelines and the surface facilities. Thus, transportation of crude oil is often threatened by flow assurance challenges. The flow assurance challenges include wax precipitation and deposition, asphaltene precipitation and deposition, scale deposition, corrosion, sands and solids deposition, etc.

The most common cause of wax solubility reduction and the consequent wax precipitation and deposition is temperature reduction. Temperature reduction could be attributed to liberation of dissolved gas from the solution and conduction of heat to the overlaying formations as the crude oil flows up the wellbore, transfer of the crude oil through surface facilities including the pipelines at ambient temperature.

Most waxy crude oils have high pour points and thus high paraffin wax content [9] with consequent low flowability where flow temperatures are about or less than the pour point. The pour point is the temperature below which the liquid loses its flow characteristics (i.e. the temperature at which the crude oil starts to exhibit semi-solid nature). The cloud point is simply the wax appearance temperature, WAT, i.e. the temperature at which the first crystal of wax appears. At the WAT, the solubility of wax in crude oil cannot be sustained causing wax to precipitate. WAT depends on wax concentration in the crude oil, wax crystallization potentials and the shear stability of wax structures [10-11]. From thermodynamics perspectives, WAT is the solid-liquid phase boundary temperature, i.e. the minimum temperature at which the solid and liquid phases co-exist in equilibrium at a fixed pressure.

When wax precipitates from crude oil, the individual wax crystal tends to disperse in the fluid. However, if the number of wax crystals increases to a threshold level or there is a presence of other nucleating substances such as asphaltene, formation fines and corrosion products in the crude oil, there may be an agglomeration of the crystals into larger particles separating out of the fluid as solid deposits.

Wax deposition in crude oil can be divided into two forms: (i) wax deposition in flow conditions and (ii) wax gelation and restart problem after shut-in. In a flow condition, three major factors influencing wax deposition are flow rate, temperature reduction and facilities surface properties [12]. The mechanisms attributable to wax deposition are molecular diffusion, shear dispersion, Brownian diffusion and gravity segregation. However, molecular diffusion is the paramount mechanism [13-14]. When production is shut down due to some emergencies, the static condition in pipelines causes temperature and wax solubility to decrease. The consequent precipitation of wax causes the formation of wax gel covering up the entire cross-section of the pipelines [15-16]. Production restart often features a remarkable problem when the ambient temperature is below the pour point.

Expensive maintenance procedures, worth billions of dollars, are carried out in preventing and remediating wax deposition [8]. One of the most commonly used methods of remediating wax deposition once it started to impede flow is pigging. In pigging, a solid object called pig, having a diameter just smaller than that of the pipe, is passed through the pipeline to scrape off the wax deposit. Proper prediction of wax deposition below the level of attaining thick and hard wax deposits is important to prevent the pigs from getting stuck in the pipelines. Correcting this would involve stopping production and replacing some portion of the pipelines. Also, remediation of wax deposition in production tubing involves using scrapers conveyed by wireline. The other deposition remediation methods are using chemicals and applying heat.

2. Literature review

Experimental (or laboratory) measures had been taken in the past to control wax precipitation; these include determining the cloud and pour points of blank crude oil, and using surfactants and polymeric pour point depressants. Surfactant is well known for improving the flow behaviour of waxy crude oil by reducing the interfacial tension of the oil at their contact, reducing the oil viscosity, and also reducing the wettability of the oil with the transportation facilities surfaces.

The application of Gemini surfactants as diesel fuel wax dispersants was studied [17]. The use of a novel surfactant for improving the flowability of Indian heavy crude oil was investigated [18]. Surfactants were observed to partition into the wax crystal structures and alter crystal morphology to spherical shapes to inhibit precipitation of wax molecules. Laboratory determination of cloud and pour points of different crude oils from the Niger Delta was carried out [19]. Cloud point of 14 to 23°C range and pour points of 2 to 14°C range were obtained. The use of bio-surfactant for wax deposition inhibition and drag reduction in crude oil pipelines was investigated [20]. The use of surfactants in improving crude oil flowability for pipeline transmission was studied [21-22]. Surfactants were observed to be effective in inhibiting wax deposition by altering oil wettability on the surface of the pipe walls. Also, surfactants blend with silica nanoparticles in inhibiting wax precipitation in crude oil was studied [23].

Polymeric wax inhibitors reduce the WAT and pour point. The traditional polymeric wax inhibitors usually have high molecular weight, long polymer chains and high thermo-stability thereby making them difficult to get decomposed and thus becoming environmentally unfriendly. Despite its environmental issues, polymeric wax inhibitors have been used and investigated; the investigation includes its synergy with nanomaterials at improving wax inhibition.

The effect of nanohybrid materials on the pour point and viscosity of waxy crude oil was studied [24]. Hydrophilic nanoparticles influence on wax inhibition was investigated [25]. Also, the effect of silicon oxide nanoparticles on wax crystallization and flow behaviour of model crude oil was studied [26]. The improved performance of nanomaterials was attributed to their ability to (i) partition into the wax crystal structures and get adsorbed onto wax crystal surfaces, (ii) alter crystal morphology through provision of a large amount of spherical-like templates onto which wax molecules can precipitate, and (iii) decrease the WAT, gelation temperature and rheological behaviour with the aid of the spherical-like templates.

Aside from investigating the potential of surfactants and their blend with silica nanoparticles in inhibiting wax precipitation in crude oil [23], extensive research has not been conducted on the potential of other metal oxide nanoparticles. Hence, this work aims at expanding the research scope by (i) determining and comparing wax precipitation in blank crude oil and same crude oils with different concentrations of metal oxide nanoparticles at temperatures below the WAT; and (ii) obtaining the more effective metal oxide nanoparticles that inhibit wax precipitation. However, prior to these, the density, specific gravity, API gravity and viscosity of the crude oil should be determined.

3. Methodology

3.1. Determination of the density, specific gravity, API gravity and viscosity of the crude oil sample

A pycnometer bottle was thoroughly cleaned, dried and weighed on a digital weighing balance before being filled to the brim with the crude oil sample and weighed again. Thus, the density of the crude oil sample ρ_o was determined by its mass (g) and volume (cm^3) values. The relative density or specific gravity of the crude oil sample γ_o was evaluated with reference to 1 g/ cm^3 density of water. The API gravity γ_{API} was thus obtained as:

$$API\ Gravity = \frac{141.5}{Specific\ gravity} - 131.5 \tag{1}$$

A thoroughly cleaned and dried Oswald viscometer was used to measure the kinematic viscosity of the crude oil sample v_O (centistoke, cSt). The crude oil sample was poured into the first arm of the viscometer tube such that the bulb on the first arm was half-filled with the oil. The oil was then sucked up to the second arm of the tube until the oil level was just at the upper reference line of the bulb on the arm. A thumb was placed on the end of the first arm to prevent the oil column in the second arm from falling. After releasing the finger, the time (efflux time) taken for the oil to flow through the upper reference line to the lower reference line of the bulb on the second arm of the tube was recorded by a stopwatch. The kinematic viscosity and the dynamic viscosity μ_O (centipoise, cP) of the crude oil, the sample was then evaluated.

3.2. Determination of cloud point (WAT) of the crude oil sample

Blank crude oil sample of 5 mL volume was poured into a test jar. The test jar was closed tightly with a cork carrying a thermometer. To facilitate effective cooling, the test jar was then placed in a methanol-filled bath inside a cloud/pour point tester. The room temperature was 35°C. As temperature drops inside the cloud point tester, the test jar was removed gently and quickly from the cloud/pour point tester at three minutes intervals for inspection. The wax appearance temperature (also called the cloud point) was recorded via the thermometer inserted into the test jar.

3.3. Determination of cloud point (WAT) of the crude oil samples with different concentrations of metal oxide nanoparticles

The procedures above were repeated for crude oil samples in which metal oxide nanoparticles were added to determine the effect of these nanoparticles on wax appearance temperature (WAT).

3.4. Wax precipitation in blank crude oil sample below wax appearance temperature

The cloud points of the blank crude oil sample were observed as 29.4°C. Hence, to determine the effect of temperature on wax precipitation in the crude oil sample, temperature points (below the WAT) outside the cloud point tester considered are 20, 10, 0, -10, and -20°C. The procedures observed in determining the cloud point (WAT) were repeated. However, a temperature drop of about 5°C was tolerated in the cloud point tester for the crude oil samples. At an appropriate temperature point in the cloud point tester, the test jar was removed gently, and the precipitates were separated quickly using a 0.35 g microfiber glass-reinforced paper filter. Using a digital weighing balance, and with the consideration of the mass of the filter, the mass of the wax precipitates was evaluated.

3.5. Wax precipitation in crude oil samples with different concentrations of metal oxide nanoparticles

The procedures above were repeated for crude oil samples in which metal oxide nanoparticles were added to determine the effect of these nanoparticles on wax precipitation at the temperatures. The range of nanoparticles concentration considered is 0.1 to 1.0g in 2 mL of crude oil sample. Here, with the consideration of the 0.35 g mass of the filter and the mass of the nanoparticles concentration, the mass of the wax precipitates was evaluated.

3.6. Determination of the more effective metal oxide nanoparticles that inhibits wax precipitation

The optimal nanoparticles concentration is the concentration above which no further reduction in wax precipitation is observed. Hence, any additional nanoparticles concentration to the optimal is considered latent and unnecessary. At the same concentrations, more effective metal oxide nanoparticles would yield minimal wax precipitation.

4. Results and discussion

4.1. Density, specific gravity, API gravity and viscosity of the crude oil sample

The volume and mass of dry and empty pycnometer bottle are 50.0 cm^3 and 19.1 g respectively, and the mass of the pycnometer bottle filled to the brim with blank crude oil sample is 65.0 g. Hence, the mass of the crude oil sample is 45.9 g, the density of the crude oil sample is 0.918 g/cm^3, and the specific gravity of the crude oil sample is 0.918. The API gravity (see Equation 1) of the crude oil sample is calculated as 22.64 indicating that the oil is a medium oil (22.3 < API Gravity < 31.1).

The viscometer constant of the Oswald viscometer is 1.3 cSt/sec. The time (efflux time) taken for the oil to flow through the upper reference line to the lower reference line of the bulb on the second arm of the viscometer tube was recorded as 60 seconds. Hence, kinematic viscosity was evaluated as 78 cSt while the dynamic viscosity was calculated as 71.60 cP. The pour and cloud points of the blank crude oil sample were observed as 12.0 and 29.4°C respectively. The properties of the crude oil sample are shown in Table 1.

Table 1. Properties of the crude oil sample

Properties	Symbol	Value	Unit	Properties	Symbol	Value	Unit
Density	ρ_o	0.918	g/cm^3	Viscosity	μ_O	71.60	cP
Specific gravity	γ_o	0.918	-	Cloud Point	WAT	29.4	°C
API gravity	γ_{API}	22.64	-				

4.2. Wax precipitation in blank crude oil

The wax precipitates evaluated at temperature points below the WAT of 29.4 °C are shown in Table 2.

Table 2. Variation of wax precipitates from blank crude oil with temperature points below the WAT

Temperature (°C)	Mass of wax precipitates (g)	Temperature (°C)	Mass of wax precipitates (g)
20	0.24	-10	0.50
10	0.28	-20	0.54
0	0.36		

4.3. Metal oxide nanoparticles effect on crude oil wax appearance temperature

Variations of wax precipitates temperature (WAT) with metal oxide nanoparticles concentration are shown in Table 3.

Table 3. Variations of wax precipitates temperature (WAT) with metal oxide nanoparticles concentration

Nanoparticles concentration (g/mL)	WAT (°C)		Nanoparticles concentration (g/mL)	WAT (°C)	
	Al_2O_3	CuO		Al_2O_3	CuO
0	29.4	29.4	0.6	20.6	21.7
0.1	26.6	28.3	0.7	20.6	21.0
0.2	23.8	26.6	0.8	20.6	21.0
0.3	22.8	25.0	0.9	20.6	21.0
0.4	21.6	23.8	1.0	20.6	21.0
0.5	21.1	22.0			

Increase in nanoparticles concentrations was observed to reduce wax precipitation temperature. This could be attributed to potential of the nanoparticles to get adsorbed onto wax crystal surfaces and change crystal morphology to spherical-like templates for improved wax stability.

4.4. Wax precipitation in crude oil samples with different concentrations of aluminium oxide nanoparticles

The wax precipitates evaluated at temperature points below the WAT are shown in Table 4.

Table 4. Variations of wax precipitates from crude oil with aluminium oxide nanoparticles at temperature points below the WAT

Nanoparticles concentration (g/L)	Temperature points below the WAT (°C)					Nanoparticles concentration (g/L)	Temperature points below the WAT (°C)				
	20	10	0	-10	-20		20	10	0	-10	-20
0	0.24	0.28	0.36	0.50	0.54	0.6	0.16	0.22	0.23	0.31	0.33
0.1	0.20	0.24	0.29	0.46	0.51	0.7	0.16	0.22	0.23	0.31	0.32
0.2	0.18	0.23	0.27	0.44	0.48	0.8	0.16	0.22	0.23	0.31	0.32
0.3	0.17	0.22	0.24	0.41	0.42	0.9	0.16	0.22	0.23	0.31	0.32
0.4	0.16	0.22	0.23	0.38	0.39	1.0	0.16	0.22	0.23	0.31	0.32
0.5	0.16	0.22	0.23	0.34	0.35						

Increase in aluminium oxide nanoparticles concentration could be explained to cause retardation in wax molecular diffusion and gravity segregation, thereby inhibiting wax precipitation. After a threshold level of nanoparticles concentration (called optimal concentration) was attained, no further decrease in wax precipitation was observed.

4.5. Wax precipitation in crude oil samples with different concentrations of copper oxide nanoparticles

The wax precipitates evaluated at temperature points below the WAT are shown in Table 5.

Table 5. Variations of wax precipitates from crude oil with copper oxide nanoparticles at a temperature points below the WAT

Nanoparticles concentration (g/L)	Temperature points below the WAT (°C)					Nanoparticles concentration (g/L)	Temperature points below the WAT (°C)				
	20	10	0	-10	-20		20	10	0	-10	-20
0	0.24	0.28	0.36	0.50	0.54	0.6	0.20	0.23	0.28	0.35	0.38
0.1	0.23	0.27	0.34	0.49	0.53	0.7	0.20	0.23	0.28	0.35	0.38
0.2	0.22	0.25	0.30	0.47	0.50	0.8	0.20	0.23	0.28	0.35	0.38
0.3	0.20	0.24	0.29	0.44	0.45	0.9	0.20	0.23	0.28	0.35	0.38
0.4	0.20	0.23	0.28	0.36	0.42	1.0	0.20	0.23	0.28	0.35	0.38
0.5	0.20	0.23	0.28	0.35	0.40						

Also, the increase in copper oxide nanoparticles concentration causes inhibition in wax precipitation unit the optimal concentration was reached.

4.6. Temperature effect on wax precipitation in crude oil samples with different concentrations of aluminium oxide nanoparticles

With reference to Table 4, the plot of wax precipitates from crude oil samples with aluminium oxide nanoparticles (NP) versus temperature points below the WAT is shown in Figure 1. Also, the plot of wax precipitates from crude oil samples with copper oxide nanoparticles (NP) versus temperature points below the WAT is shown in Figure 2.

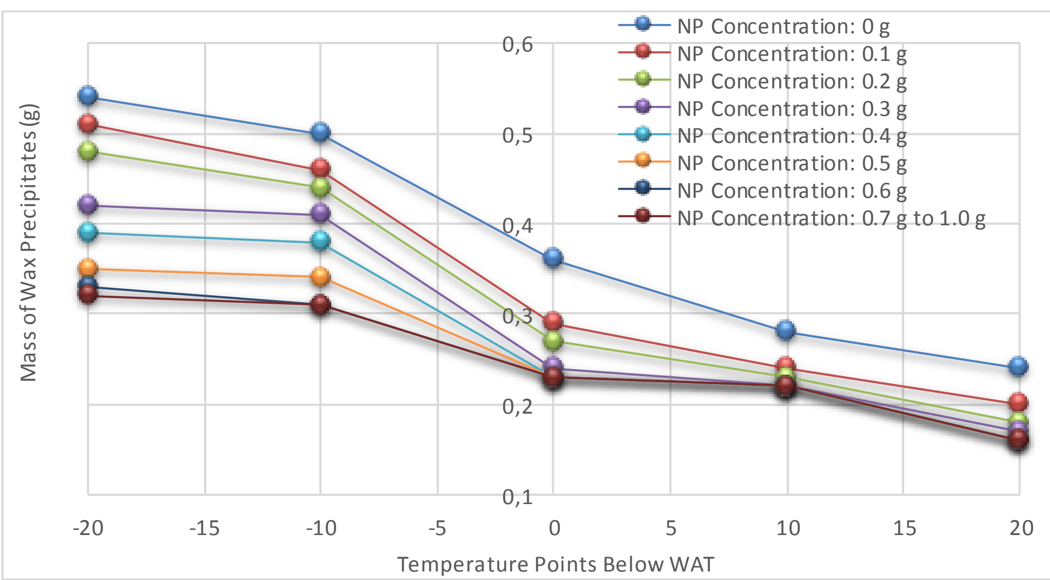

Figure 1 Plot of wax precipitates from crude oil samples with aluminium oxide nanoparticles versus temperature points below the WAT

With temperature decrease from room temperature to the onset of WAT, the solubility of wax in crude oil was sustained. However, just below the WAT, the solubility of wax could no longer be sustained, causing wax to precipitate. And further decrease in temperature causes more wax precipitation.

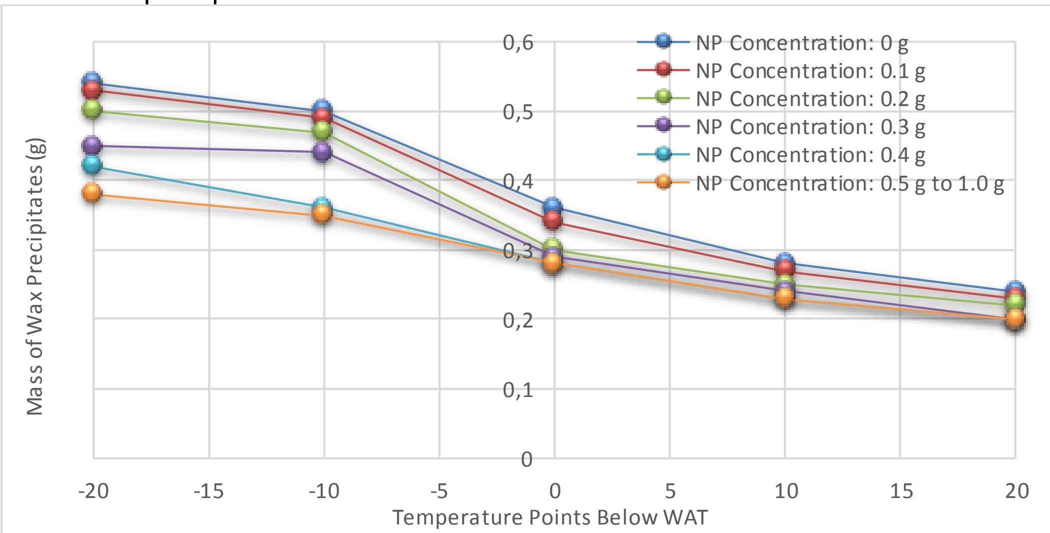

Figure 6. Plot of wax precipitates from crude oil samples with copper oxide nanoparticles versus temperature points below the WAT

4.7. Determining the more effective metal oxide nanoparticles inhibiting wax precipitation

From Tables 4 and 5 showing the variations of wax precipitates from crude oil with nanoparticles at temperature points below the WAT, mass at temperature points below the WAT versus nanoparticles concentration, the optimal metal oxide nanoparticles concentrations inhibiting wax precipitation can be obtained.

At 20, 10, 0, -10 and -20°C, the optimal aluminium oxide nanoparticles concentrations inhibiting wax precipitation are 0.4, 0.3, 0.4, 0.6 and 0.7g with corresponding minimal wax precipitations of 0.16, 0.22, 0.23, 0.31 and 0.32 g. However, at the respective temperatures, the optimal copper oxide nanoparticles concentrations inhibiting wax precipitation are 0.3, 0.4, 0.4, 0.5 and 0.6g with corresponding minimal wax precipitations of 0.20, 0.23, 0.28, 0.35 and 0.38 g.

Hence, this study shows that aluminium oxide nanoparticles are more effective than copper oxide nanoparticles in reducing wax precipitation and deposition in crude oil. Thus, aluminium oxide nanoparticles are established to have more affinity than copper oxide nanoparticles in dissolving and keeping wax stable in crude oil.

5. Conclusions

The decrease in temperature increases wax precipitation. Reducing temperature of blank crude oil from room temperature of 35°C to 20°C and -20°C in the cloud point tester causes wax precipitation to increase from 0.24 g to 0.54 g respectively.

For both metal oxide nanoparticles, the increase in nanoparticles concentrations reduces wax precipitation. At 20°C, wax precipitation reduces from 0.24 g in blank crude oil to 0.16 g with 0.40 g aluminium oxide nanoparticles addition. At 20, 10, 0, -10 and -20°C, the optimal aluminium oxide nanoparticles concentrations inhibiting wax precipitation are 0.4, 0.3, 0.4, 0.6 and 0.7g with corresponding minimal wax precipitations of 0.16, 0.22, 0.23, 0.31 and 0.32 g.

At the respective temperatures, the optimal copper oxide nanoparticles concentrations inhibiting wax precipitation are 0.3, 0.4, 0.4, 0.5 and 0.6g with corresponding minimal wax precipitations of 0.20, 0.23, 0.28, 0.35 and 0.38 g. Therefore, this study shows that aluminium oxide nanoparticles are more effective than copper oxide nanoparticles in reducing wax precipitation and deposition in crude oil.

Recommendations

Investigating the potentials of other metal oxide nanoparticles is recommended for further studies.

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