Contents lists available at ScienceDirect





Tribology International

journal homepage: www.elsevier.com/locate/triboint

Dispersing mechanism and tribological performance of vegetable oil-based CNT nanofluids with different surfactants



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ARTICLE INFO

Keywords: Surfactant Dispersion mechanism CNT nanofluids Tribological performance

ABSTRACT

CNTs are commonly used nanoparticles in NMQL for their excellent heat transfer enhancement performance. However, winding and conglobation are constraints against their tribological performance, these constraints can be solved by surfactants. However, the influences of surfactant type and dispersing mechanism on the dispersion effect of CNTs have not been studied systemically. Consequently, this study analyzed the dispersing mechanism of different surfactants and evaluated the dispersion stability and tribological performances of PPO-based CNT nanofluids. Results showed that nanofluids with APE-10 obtain the highest viscosity, lowest friction coefficient, minimum roughness value and favorable surface morphology, thus indicating their excellent dispersion stability and tribological performance. Further, different experimental evaluations confirm that APE-10 is the optimal dispersant of CNT nanofluids.

1. Introduction

Pouring grinding, which has a large flow rate of grinding fluid, increases the cost for grinding fluid disposal, consumes substantial grinding fluid, and causes considerable environmental damages [1,2]. In this context, a new precision-machining technique with high efficiency, low consumption, and clean low-carbon consumption, that is, NMQL, has been developed [3-5]. Solid nanoparticles are added to base oil to prepare nanofluids, which can solve the technological bottleneck of poor heat exchange performance in the traditional MQL grinding by exploiting the excellent heat transfer enhancement of nanoparticles [6-8]. Li et al. [9] investigated into the effect of NMQL on the temperatures in surface grinding and fonud that CNT nanofluid results in the lowest grinding temperature of 110.7 °C. Then, these researchers developed a mathematical model for convective heat transfer coefficient which to some extent explained the experimental result. Nanoparticles are conducive to improving the lubrication performance on grinding wheel/workpiece and wheel/cutting interfaces in the MQL [10,11], thereby improving machining accuracy and surface quality significantly [12,13]. Nanoparticles can further increase the

tribological performance in the grinding zone [14–16]. Ge et al. [17] explored the tribological behaviors of new greases added with oleophilic TiO₂ and SiO₂ nanoparticles. These researchers found that a layer of protective oil film is generated by the deposited Ti and Si or metallic oxide, which can reduce friction and improve tribological properties. Qu et al. [18] demonstrated that Cu microparticles can improve the friction-reduction and antiwear properties of sunflower seed oil, and the synergistic lubrication effect is produced to explain the lubrication mechanisms. Zhang et al. [19] explored the lubrication performance of the NMQL by adding mixed nanoparticles (MoS₂/CNTs). The results showed that the MoS₂/CNT (optimal mixing ratio is 2:1.) hybrid nanoparticles achieve a better lubrication effect than single nanoparticles. Vegetable oil is a nontoxic, biodegradable, and environment-friendly renewable resource [20]. This resource has low or zero damage to the environment and operators and has an excellent lubrication performance [21]. Therefore, vegetable oil can replace the traditional mineral oil as the base oil of lubricating fluid [22,23]. Guo et al. [24] used castor oil as the base oil and individually mixed it with six other types of vegetable oils at a ratio of 1:1; each mixture was obtained as the base oil for MQL grinding. The results indicated that the comprehensive

https://doi.org/10.1016/j.triboint.2018.10.025

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Received 1 July 2018; Received in revised form 19 August 2018; Accepted 19 October 2018 Available online 21 October 2018

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Nomenclature and abbreviation		A_{dm}	Hamaker constant of dispersive media
		A_p	Hamaker constant of particles
NMQL	nanofluid minimum quantity lubrication	Α	Hamaker constant of particles in the medium
MQL	minimum quantity lubrication	A_s	Hamaker constant of surfactants adsorbed on the surface
MWCNT	multi-walled carbon nanotube		of the particles
SWCNT	single-walled carbon nanotube	Δ	shortest distance between the adsorption layers of two
CNT	carbon nanotube		isometric spheric particles
PPO	pure palm oil	δ	adsorption layer thickness
Nanoflui	ds fluid containing nanometer-sized particles	а	effective radius of spheric particles
Mr	relative molecular mass	V_R^{S}	space repulsive potential energy
HLB	hydrophilic-lipophilic balance	$V_R^{\ heta}$	entropy repulsive potential energy
NPE	nonylphenol polyoxyethylene ether	k	Boltzmann constant
OPE	octyl phenol polyoxyethylene ether	N_S	number of adsorbed molecules on the surface area of the
DPE	dodecyl polyoxyethylene ether		unit
DNPE	dinonyl phenol polyoxyethylene ether	H_0	shortest distance between the surfaces of two spheric
APE-10	alkylphenol polyoxyethylene ether-10		particles
CTAB	cetyl trimethyl ammonium bromide	$\boldsymbol{ heta}_{\infty}$	extent of the surface to be covered by the molecule when
OP-10	octyl phenol polyoxyethylene ether-10		the two surface distances are $H_0 = \infty$
SDBS	sodium dodecyl benzene sulfonate	1	length of the adsorbed surfactant molecule
SDS	sodium dodecyl sulfate	V_R^E	elastic repulsive potential energy
TTAB	tetradecyl trimethyl ammonium bromide	G	elastic modulus of the adsorption layer
SEM	scanning electron microscope	V_R^O	osmotic repulsive potential energy
TEM	transmission electron microscope	A_2	second virial coefficient
[EAMIM]	BF ₄ functionalized 1-ethoxycarbonylmethyl-3-methylimi-	C_2	concentration of surfactants in the adsorbed layer
	dazolium tetrafluoroborate	Т	absolute temperature
V_A	van der Waals suction potential energy	CMC	critical micelle concentration
V_R	electrostatic repulsive potential energy		

lubrication performance of mixed oil is superior to that of castor oil, and soybean/castor oil exhibits the optimal performance and surface quality. Wang et al. [25] explored the lubrication performance of seven vegetable oils in the MQL grinding by comparing these oils with traditional pouring lubrication. These researchers found that vegetable oils possess an excellent lubrication performance, as manifested by low friction coefficient and grinding wheel wear; the results indicated that palm oil has the most excellent lubrication performance. Under ambient temperature, the heat conductivity coefficient of CNTs is 3000 W/m·K, which is a thousand times higher than that of water. For the solid materials of the same type and mass, a small volume indicates small grain size, large specific surface area of materials, and high heat-conducting properties [26,27]. The thermal conductivity of several common materials is listed in Table 1 [28]. In addition, scholars have also done research on grinding wheel to reduce the grinding temperature and improve the lubrication and cooling performance of grinding process. Mao et al. [29] proposed a spiral orderly distributed fiber tool and carried out the grinding experiment. The results indicated that the useful flow of cutting fluid for the spiral orderly distributed fiber tool is increased obviously. Miao et al. [30] investigated the influence of graphite addition on bonding properties of the abrasive layer and decided an optimized graphite content of 10 wt%, at which the porosity of abrasive layer and the bending strength was favorable to meet the high-speed grinding requirements.

CNTs have a small size, a large specific surface area, and a draw ratio. Their surface energy and tension are also significantly higher than those of conventional materials. CNTs are easy to wind and conglomerate and then can form large aggregates given the robust electrostatic and van der Waals force among molecules or atoms on the surface, as illustrated in Fig. 1. The conglomeration of CNTs can cause incomplete oil membrane and large winding accumulations on the frictional pair surface; thus, it cannot develop an effective rolling and serve as "microbearing" similar to several spherical nanoparticles. These abovementioned features determine the limited friction-reduction capability of CNTs [31]. Wang et al. [32] introduced the lubrication properties of different vegetable oil-based nanofluids by comparing frictional tests and grinding experiments. The results showed that all the nanoparticles can improve the lubrication performance of the base oil, in which the friction coefficient of CNT nanofluids fluctuates with time, and the wear performance is poor. Xie et al. [33] added different nanoparticles to the base liquid to prepare nanofluids. These researchers studied the influences of particle size, volume concentration, pretreatment process, additives, and the physical properties of the base liquid on heat conduction and proposed promising approaches to optimizing the thermal conductivity of nanofluids. Hu et al. [34] added sorbitol monostearate and copper stearate as dispersing agents to 500SN oil that contained amorphous ferric oxide nanoparticles and found that the dispersing agents play important roles in improving the tribological properties; furthermore, copper stearate is markedly superior to sorbitol monostearate. The stability and homogeneity of nanofluids improve the thermal conductivity significantly. A stable uniform dispersion of CNTs is necessary to restrain their winding and conglomeration and optimize their performance [35].

Scholars have conducted numerous studies to explore the dispersing effects of various surfactants on CNTs. Duan et al. [36] reported the dispersion of CNTs with SDS by molecular mechanic simulations from an energy perspective. These authors revealed the aggregation morphologies of SDS on the surface of CNTs and the effect of the

Table 1 Thermal conductivity of several common materials.

Material	Engine oil	Water	Copper oxide	Alumina	Silicon	Aluminum	Copper	Diamond	CNTs
Thermal Conductivity/W/(m·K)	0.145	0.613	19.6	40	148	237	401	2300	3000



Fig. 1. SEM images of CNTs.

diameter of CNTs on the adsorption density. Moore et al. [37] suspended SWCNT in water-bearing media by using various anionic, cationic, and non-ionic surfactants and polymers. These researchers compared the capability of the SWCNT suspension of surfactants and the qualities of absorption and fluorescence spectra. The results demonstrated that ionic surfactant SDBS has gained the optimal-resolution spectral characteristics. The non-ionic surfactant contains a high molecular mass of surfactant to suspend substantial SWCNT and gain obvious spectral characteristics. Rastogi et al. [38] reported a comparative analysis of the dispersion of MWCNTs with four surfactants, namely, Triton X-100, Tween 20, Tween 80, and SDS. Among the four surfactants, Triton X-100 and SDS provided the maximum and minimum dispersions, respectively. The experimentally observed trend of the dispersing power of surfactants was consistent with their chemical structures. Estellé et al. [39] measured the viscosity and thermal conductivity of water-based CNT nanofluids, which were stabilized by lignin as a surfactant. In contrast to SDBS, lignin reduced the viscosity and shear-thinning behavior of nanofluids at a high-volume fraction, without penalizing thermal conductivity enhancement.

Scholars have also studied the performance of other nanofluids under the condition of surfactant. Mao et al. [40] studied the effect of SDBS on the suspension stability of Al_2O_3 nanofluids and analyzed the dispersion morphology of Al_2O_3 nanoparticles in a disperse system. These researchers found that large aggregates appear when no surfactant is applied in the disperse system, and the Al_2O_3 nanoparticles are uniformly dispersed in the disperse system by applying the surfactant. Guo et al. [41] investigated the tribological properties of oil-based nanofluids using different surfactants. The results revealed that phosphinate ionic liquid, as the surfactant, maintains long-term stability when mixed with ceramic nanoparticles. SEM studies showed that the film formed on the disk surface may enhance the tribological performance when using oleic acid as the surfactant. Behera et al. [42] studied the correlation between the spreading behavior, surface tension, and surface energy of Al_2O_3 nanofluids added with different surfactants. The optimal wetting behavior was observed when the nanofluids were added with a non-ionic surfactant. Machining experiments with Inconel 718 using the aforementioned nanofluids indicated a reduction in friction coefficient.

The literature index demonstrated that scholars have observed the macroscopic dispersion effects of CNTs and analyzed the particle sizes of CNT aggregates using laser granulometer and other instruments. The effects of different surfactants on the dispersion stability of CNTs were studied through SEM and TEM. However, the influence mechanisms of different surfactants on CNTs are diverse, which is caused by their different chemical molecular structures and physical properties. Currently, only a few studies compare the tribological performances and analyze the corresponding mechanism of CNT nanofluids added with different types of surfactants. Thus, the surfactant that can contribute the optimal CNT dispersion, the CNT nanofluids with the type of surfactant with the optimal tribological performance, and if an essential relationship with the dispersion stability exists cannot be determined. The experimental phenomenon is also difficult to capture by evaluating the tribological performance of lubricating fluid by grinding tests, and evaluating the lubrication performance of the lubricating fluid by frictional testing is a conventional method. The working conditions for frictional and grinding tests are different, but the frictional test can be used to simulate the conditions of grinding test and represent the tribological properties of grinding fluid, thus enabling this test to guide the actual industrial production. At present, rare studies on this aspect have not been reported.

The work and aims of this study are as follows.

- To determine the influences of different surfactants on a CNT-dispersing mechanism
- To observe the dispersing stabilities of different surfactants through a static method
- To evaluate the tribological performances of nanofluids added with six types of surfactants and analyze the friction-reduction and antiwear principles using a frictional tester
- To explore and determine the surfactant that is the most suitable dispersing agent for CNT nanofluids

2. Dispersion mechanism analysis and comparison of surfactants



CNTs are coaxial circular tubes composed of a monolayer to

Fig. 2. Space barrier of surfactant adsorption.

multilayer carbon atoms in a hexagonal arrangement. Carbon atoms in CNTs hybridized by SP^2 have a larger S orbit than those hybridized by SP^3 , thus exhibiting high modulus and strength [43]. CNTs possess favorable lubrication performance and wearing resistance considering their unique tube structure and extremely high tenacity. However, CNTs are characterized by their tubular, easy-winding, and conglobation characteristics, which limit their applications. The addition of surfactants to CNT nanofluids can relieve the winding and conglomeration of CNTs effectively, thereby improving the CNT dispersion effect. Different surfactants have various dispersing mechanisms to CNTs.

2.1. Space barriers of CNT-PPO and CNT-CNT interfaces

CNT surface is classified as a high-energy surface, and palm oil possesses favorable polarity given its high triglyceride content. Therefore, palm oil cannot spread over the CNT surface well. Moreover, CNTs are easy to wind and conglomerate, thus resulting in the failure of a uniform CNT dispersion in PPO, as depicted in Fig. 2a. Surfactant molecules with special groups can be adsorbed onto the CNT surface or PPO strongly. Space barrier is formed in this way to assure uniform dispersion of CNTs in PPO after adding a surfactant, and the schematic of a space barrier is demonstrated in Fig. 2b.

The main role of surfactant as the dispersing agent is to prevent the re-conglomeration of dispersed solid particles. The disperse system is a thermodynamic instability system given its large surface area and high surface energy. The surfactant adsorbed onto the CNT-PPO interface weakens the tension of the CNT-PPO interface, thus decreasing the surface free energy significantly and relieving the trend of mutual conglomeration. In addition, the adsorption layer on the CNT surface can be thickened by the surfactant adsorption, and the formed space barrier can hinder the conglomeration of particles [44]. If the surfactant is classified as an ionic-type surfactant, then the adsorption of surfactant also causes charges to the surface to generate an electrostatic repulsion barrier, which can hinder the conglomeration of particles. According to DLVO theory, the stability of the disperse system is determined by the sum of the van der Waals suction potential energy V_A and the electrostatic repulsive potential energy V_R of the particles as follows [45,46]:

$$V = V_A + V_R \tag{1}$$

$$V_R = 0 \tag{2}$$

In the disperse system of non-aqueous media, the electrostatic repulsion barrier can be neglected given the low dielectric constant, and the dispersion stability of solid particles is mainly determined by V_A .

 V_A depends on particle size, particle shape, distance among particles, and nature of dispersed phase and medium. For two pellets with equal diameter, V_A complies with the following formula:

$$V_{A} = -\frac{A}{12} \left[\frac{1}{(H_{0}/2a)^{2} + (H_{0}/a)} + \frac{1}{(H_{0}/2a)^{2} + (H_{0}/a) + 1} + 2\ln\frac{(H_{0}/2a)^{2} + (H_{0}/a)}{(H_{0}/2a)^{2} + (H_{0}/a) + 1} \right]$$
(3)

where *A* is the Hamaker constant, H_0 is the shortest distance between the surfaces of two spheric particles, and *a* is the effective radius of the spheric particles. The Hamaker constants of particles and dispersive media are A_p and A_{dm} , respectively. The Hamaker constant of particles *A* in the medium will change and can be calculated using the following equation:

$$A = (A_{dm}^{1/2} - A_p^{1/2})^2 \tag{4}$$

Surfactants adsorbed on the surface of the particles will form an adsorption layer with a thickness of δ , and the Hamaker constant of surfactants is A_s . When the shortest distance between two isometric spheric particles is Δ , the expression of V_A is as follows:

$$V_{A} = -\frac{1}{12} \left[(A_{p}^{1/2} - A_{s}^{1/2})^{2} \left(\frac{a+\delta}{\Delta} \right) + (A_{s}^{1/2} - A_{dm}^{1/2})^{2} \frac{a}{\Delta + 2\delta} + \frac{4a(A_{p}^{1/2} - A_{s}^{1/2})^{2}(A_{s}^{1/2} - A_{dm}^{1/2})^{2}a+\delta}{\Delta + \delta - 2a+\delta} \right]$$
(5)

Formula (5) implies that the absolute value of V_A decreases with the increase in δ ; that is, the adsorption layer can reduce the suction potential energy and increase the stability. The absolute value of V_A increases with *a*; in particular, the suction potential energy increases, and the stability becomes poor with the increase in *a*.

In addition to affecting V_A , the surfactant can also produce a barrier called a space repulsive potential energy V_R^s considering the formation of the adsorbed layer and solvation effect. The nature of the adsorbed layer indicates three parts of V_R^s , namely, V_R^θ , V_R^E , and V_R^o .

Space repulsive energy can be written as

$$V_R^S = V_R^\theta + V_R^E + V_R^O \tag{6}$$

The first part is the entropy repulsive potential energy V_R^{θ} , which can be expressed as follows:

$$V_R^{\theta} = N_S k T \theta_{\infty} \left(1 - \frac{H_0}{l} \right)$$
(7)

where N_S is the number of adsorbed molecules on the surface area of the unit, k is the Boltzmann constant, θ_{∞} is the extent of the surface to be covered by the molecule when the two surface distances are $H_0 = \infty$, l is the length of the adsorbed surfactant molecule, and T is the absolute temperature.

The surfactant adsorbed on the solid surface is used as a rigid rod, and the degree of activity freedom of a steel-rod surfactant will be reduced when the two surfaces draw close, thus producing the entropy repulsive potential energy.

The second part is the elastic repulsive potential energy V_R^E , which can be expressed as follows:

$$V_R^E = 0.75G \left(\delta - \frac{H_0}{2}\right)^{5/2} (a+\delta)^{1/2}$$
(8)

where *G* is the elastic modulus of the adsorption layer, δ is the adsorption layer thickness, *H*₀ is the shortest distance between the surfaces of two spheric particles, and *a* is the effective radius of the spheric particles. The surfactants are elastomers; thus, *V*_R^{*E*} will be generated when the adsorbed layer is compressed.

The third part is the osmotic repulsive potential energy V_R^{O} , which can be expressed as follows:

$$V_R^0 = \frac{4}{3}\pi kT A_2 C_2 \left(\delta - \frac{H_0}{2}\right)^2 \left(3a + 2\delta + \frac{H_0}{2}\right)$$
(9)

where *k* is the Boltzmann constant, *T* is the absolute temperature, A_2 is the second virial coefficient, C_2 is the concentration of surfactants in the adsorbed layer, δ is the adsorption layer thickness, H_0 is the shortest distance between the surfaces of two spheric particles, and *a* is the effective radius of the spheric particles.

When the adsorption layer overlaps, the surfactant concentration will increase in the overlapped region, and the chemical potential difference is due to the concentration difference between the overlap area and the adsorbed layer; this difference leads to the repulsive potential energy V_R^{O} .

2.2. Reversed micellization

The micelle formation of surfactants in the solution closely resembles the surfactant adsorption at an interface. The state of surfactant molecules will change when a certain concentration is reached. Under this circumstance, a certain number of surfactant molecules in the solution will create an aggregation in the form of a polar group

proceeding to opposite directions, and the aggregation is a micelle, which can effectively increase the space repulsion barriers [45]. The number of micelles increases with the further increase in surfactant concentration while keeping the size of each micelle and the number of molecules in each micelle constant, and the entire process is exhibited in Fig. 3. A certain surfactant concentration is required to form micelles, and the certain concentration is described as a critical micelle concentration (CMC). The micelle formation will cause sudden changes in the surfactant properties. The CMC is an important property of surfactant and can be used as an evaluation index of the surface activity of the surfactant. A low CMC indicates a low concentration required to form micelles and a low concentration for the interface to reach the saturated adsorption of the monomolecular laver. Therefore, a low concentration can significantly change the internal and surface properties of the system; that is, surfactant performs effectively under low micelle concentration [45,47].

Surfactants can also form micelles in a non-aqueous solution. Such aggregate, with hydrophilic groups as the internal kernel, is called reversed micelle (W/O micelle) and has opposite structures with micelles in the aqueous solution. The association of surfactant in a nonpolar solvent is the consequence of forming a reversed micelle through dipole–dipole between two molecules and ion interaction. Reversed micellization is different from micelles formed by surfactant in water through hydrophobic interaction; This process is complicated, as displayed in Fig. 4. The reversed micellization further increases the space barrier and improves the dispersion stability of CNTs.

Several special groups and chemical bonds in surfactant can influence the CMC differently. The -O- and -OH polar groups in the molecular hydrocarbon chain of surfactant will decrease the critical reversed micelle concentration of surfactant significantly [45].

2.3. Nature of the hydrocarbon chain

The hydrophilicity of the hydrophilic group is negatively related to the length of the hydrocarbon chain. A strong relative hydrophilicity of hydrophilic groups implies a strong tendency to escape from oil, easiness to be adsorbed onto the solid surface, and a high adsorption capacity. Surfactants with significantly different properties also conform to the abovementioned law [45]. For surfactants that belong to homolog, the surface adsorption capacity will increase slightly with the growth of methyne $-CH_2$ in the hydrocarbon chain length [48].

The hydrocarbon chain length of ionic surfactant can influence its hydrophilicity and lipophilicity; that is, a long hydrocarbon chain leads to a strong lipophilicity. However, such a relationship is sensitive to the hydrophilicity of hydrophilic groups. Given strong lipophilicity, the adsorption capacity on CNT–PPO interface enhances, and the absorption stability improves. However, the long chain will be curled up when the carbon number in the straight alkane chain of the lipophilic group increases up to 16, thus also increasing the single-molecule cross-sectional area of the surfactant. Consequently, the saturated adsorption capacity of the surfactant decreases, and the adsorption layer becomes loose, thereby indicating the declining adsorption capacity of the surfactant [45].

2.4. London force

An instantaneous relative displacement between electron cloud and atomic nucleus frequently occurs given the ceaseless movement of electrons and the continuous vibration of an atomic nucleus in molecules; thus, an instantaneous dipole is produced. The instantaneous dipole exists briefly but continuously. The molecular interaction force generated by the instantaneous dipole is called the London force, and London force constantly occurs on the surfactant and solid surface. The significance of London force lies not only in its independent adsorption mechanism but also in its supplementation mechanism of other adsorption types [45]. London force increases with the molecular weight of surfactant. London force is also related to molecular deformability, ionization potential, and molecular distance.

2.5. Synergism

Mixed surfactant possesses excellent performance, is prepared by two or several types of surfactants, and has an excellent performance [45,49]. The physicochemical properties of the mixed surfactant solution change significantly compared with those of the independent surfactant, which is called synergism or called synergistic effect. The surface activity of mixed surfactant is superior to that of each component, and several of its properties may even be beyond the capability of the original components. The mixed surfactant prepared by homolog can be approximately viewed as the ideal solution, and micelle is viewed as the ideal micelle [50,51].

3. Experimental

3.1. Experimental setup and conditions

Frictional tests under different lubricants were performed using a ball-on-disc rotation sliding tribotester (UMT-3 Instruments). Friction coefficient was recorded in a computer connected to the testing platform automatically. The experimental apparatus and principle are presented in Fig. 5. To conform to the grinding conditions, a white alundum ball and a stainless steel disc were selected to be the friction pair in the experiment. The rotation sliding between the disc and white alundum ball approximately simulated the lubrication state between abrasive particles and workpiece surface in the surface grinding experiment. The experimental parameters are listed in Table 2.

Six lubricating fluids (six types of surfactants + CNT nanofluids) were used in the frictional test, and the PPO was selected as the base oil, according to [52]. The control variate method of the test was adopted. In each test, only the type of lubricant was changed, whereas other conditions remained the same. The overall test design is displayed in Table 3. Each frictional test was performed thrice to assure a high test accuracy. After each test, the white alundum ball and disc were cleaned by acetone and absolute ethyl alcohol to eliminate impurity residues



Fig. 3. Formation process of micelles.



Fig. 4. Space barrier of reversed micelles.

(e.g., lubricating fluid and cutting debris) on the sample for the convenience of further measurements.

The SEM images and surface roughness value of disc and the material removal rate were measured to analyze the tribological characteristics of CNT nanofluids added with different surfactants. Wear samples were processed by 10 min ultrasonic cleaning in acetone and then cleaned by absolute ethyl alcohol to reduce measurement error. Finally, the samples were dried, packed in bags, and labeled. The viscosity of CNT nanofluids with different surfactants under room temperature (approximately 23 °C) was measured using a DV2TLV digital viscometer. The test and analysis equipment is illustrated in Fig. 6.

4. Materials

An AISI304 steel disc and a white alundum ball were used for the two friction pairs. The element composition and properties of the AISI304 disc are listed in Tables 4 and 5, correspondingly. The basic properties of the white alundum ball are listed in Table 6.

In the experiment, PPO was used as the basic lubricating oil and CNTs as the nanoparticles. The main properties of PPO and CNTs are listed in Table 7. Surfactants with different basic groups and

Table 2	
Tribotesting	parameters.

Condition	Value
Test temperature (°C)	22 ± 5
Test duration (s)	1800
Load (N)	20
Rotating speed (r/min)	50

Гable	3	

Sample	Lubricating fluid composition
1–1	PPO+2 wt% (CNTs)+3 wt% (APE-10)
1–2	PPO+2 wt% (CNTs)+3 wt% (CTAB)
1–3	PPO+2 wt% (CNTs)+3 wt% (OP-10)
1-4	PPO+2 wt% (CNTs)+3 wt% (SDBS)
1–5	PPO+2 wt% (CNTs)+3 wt% (SDS)
1–6	PPO+2 wt% (CNTs)+3 wt% (TTAB)

hydrocarbon chain lengths were applied. The main properties of the



Fig. 5. UMT-3 Tribometer 1. lubricant, 2. grind disc, 3. white alundum ball, 4. cage, 5. force sensor, 6. fixed block, 7. Z-axis slide rail, 8. synchronous pulley, 9. belt.



Fig. 6. Diagram of the test and analysis equipment.

surfactants are summarized in Table 8.

4. Results and discussion

4.1. Stability persistence

Variations in CNT nanofluids with different surfactants under static conditions were observed and recorded, as depicted in Fig. 7. Prepared CNT nanofluids with different surfactants were processed through a 10 min ultrasonic vibration in an ultrasonic cleaning machine to ensure the same initial dispersion state. After putting static for 1 h, the CNT nanofluids with SDS showed obvious layering; their bottom layer was the CNT aggregation layer, the middle was the PPO layer with few CNTs, and the top was the SDS particulate layer. SDS is a particulate surfactant with high HLB and poor oil solubility, and abundant SDS particles are accumulated on the top layer rather than dispersing [53]. After 3 h of being static, the CNT nanofluids with SDBS began to demonstrate CNT aggregation settlement, and the color lightened up from the bottom to the top of the tube. The CNT nanofluids with CTAB began to develop a flocculent CNT aggregation system in the upper part and abundant CNT aggregation settlement in the lower part. After 5 h of being static, the CNT nanofluids with SDS, SDBS, and CTAB continued to aggregate and deposit, and the layering phenomenon intensified. However, no obvious changes were observed in the other groups of nanofluids. After 21 h of being static, the CNT nanofluids with APE-10 and OP-10 developed extremely thin transparent layers on the liquid surface; these transparent layers might be a small amount of mixture of surfactants and palm oil. No obvious changes occurred in the other groups of nanofluids. After 45 h of being static, none of the nanofluids

Table 5

Properties of A	ISI304 material.
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Physical properties	
Melting point	1398 °C-1454 °C
Density	7850 kg/m ³
Elastic modulus at 20 °C	199000 MPa
Specific heat capacity	500 J/(kg·K)
Thermal conductivity at 20 °C	15 W/(m·K)
Mechanical properties	
Inner stress	205 N/mm^2
Tensile strength	520 N/mm^2
Hardness	90 HRB
Elongation	40%

Table 6

Basic properties of the white alundum ball.

Model	Grain size	Hardness	Binding agent	Ball diameter
WA80H12V	240 mesh	medium	ceramic	9.5 mm

had developed marked changes. Therefore, APE-10(> 45 h), OP-10(> 45 h), and TTAB(> 45 h) were the surfactants with a high dispersion stability under extended time statics.

4.2. Effects of surfactants on the viscosity of CNT nanofluids

The dispersing mechanisms of the different surfactants in the CNT

Table	4
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Element composition	of AISI304 material.
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F								
Element	С	Si	Mn	Cr	Ni	S	Р	Fe
Weight (%)	≤0.08	≤1.00	≤2.00	18.0–20.0	8.0–10.5	≤0.03	≤0.035	Remainder

Table 7 Main properties of CNTs and PPC

Main	properties	of	CNTs	and	PPO.

Properties of nanoparticles				
Nanoparticles	CNTs			
Size	50 nm			
Purity	99.0%			
Color	black			
Density	2.1 g/cm^3			
Properties of base fluid				
Base oil	PPO			
Color	yellow			
Density at 20 °C	0.93 g/ml ³			
Viscosity at 20 °C	56.7 MPa s			
Flashpoint	165 °C			
Acid value	0.16 mg/g			
Saponification	195.07 mg/g			
Main fatty acid	42.02% oleic acid, 40.24% palmitic acid, 12.32%			
composition	linoleic acid, 4.27% stearic acid			

nanofluids were discussed by static observation and viscosity comparison. The prepared CNT nanofluids with different surfactants were dispersed through a 10 min ultrasonic vibration in the ultrasonic cleaning machine, and viscosity was measured using the DV2TLV digital viscometer. The test parameters were set as follows: 75% torque, 1 min measurement time, and viscosity data collected every 1 s. Finally, the viscosity average throughout the entire measurement was obtained.

The viscosities of the CNT nanofluids added with different surfactants under room temperature are demonstrated in Fig. 8. Fig. 8 illustrates that adding surfactant can increase the viscosity of the CNT nanofluids. Different surfactants increased the viscosity of the CNT

Table 8

nanofluids to different extents. The order of viscosity of lubricating fluids was (PPO + CNTs + APE-10) > (PPO + CNTs + CTAB) > (PPO + CNTs + SDS) > (PPO + CNTs + TTAB) > (PPO + CNTs + OP-10) > (PPO + CNTs + SDBS) > (PPO + CNTs) > (PPO). The comparison of the measurement results with frictional test and static observation showed that the nanofluids with APE-10 and CTAB exhibit improved dispersion stability and tribological properties. APE-10 increased the viscosity of nanofluids significantly because it is a mixed surfactant; that is, different surfactant molecules had high-efficiency "synergism" and were adsorbed onto the CNT–PPO interface [45,49].

CTAB could increase the viscosity of nanofluids more significantly than the TTAB; this phenomenon could be explained by the following aspects. CTAB contains two hydrophilic groups (amidogens and halogen -Br). With strong hydrophilicity, CTAB shows a high tendency to escape from oil and is easy to be adsorbed onto the CNT surface [45]. Thus, the adsorption capacity is high, which is conducive to a uniform CNT dispersion and a high nanofluid viscosity. In addition, the carbon chain is longer in the lipophilic groups of CTAB than in TTAB, and the longer chain will not be curled up in a short time. CTAB is adsorbed onto CNTs, thereby increasing the distance between CNTs compared with that under TTAB adsorption and improving the CNT dispersion effect [45,46].

Nanofluids with SDS achieved the third highest viscosity but also poorer dispersion stability. The reasons for this result might be as follows: SDS is a powdery surfactant with a high HLB value; therefore, the oil solubility of SDS is poor, and the dispersion effect of CNT nanofluids after adding SDS deteriorates [53]. Moreover, serious layering phenomena, namely, CNT deposited at the bottom of the lubricating fluid, PPO occupied the middle layer, and SDS particles accumulated at the





Fig. 7. Static observation results of nanofluids with different surfactants.



Fig. 8. Viscosity of nanofluids with different surfactants.

top layer, was developed, thus resulting in high viscosity. The low viscosity of the nanofluids with OP-10 might be caused by the weak London force of OP-10 molecules on the CNT surface [45]. The rotor rotation of viscometer in the measurement significantly damaged the adsorption of OP-10 considering the strong external disturbance and the weak London force of OP-10 molecules on the CNT surface.

4.3. Effects of surfactants on the *friction-reduction performance* of CNT nanofluids

The utilization of surfactant could increase the dispersion stability of CNTs in PPO. However, different surfactants caused various friction coefficients. Therefore, the possible reasons and mechanisms of actions related to the friction-reduction effects of CNT nanofluids with different surfactants must be discussed. The experimental results of the friction coefficient and viscosity were highly correlated. CNT nanofluids with surfactant possessed high viscosity; thus, a low friction coefficient was obtained. With the increase in viscosity, the colloidal force, Brownian movement, and viscosity force among nanofluid molecules increased accordingly, thereby benefitting the formation of oil membrane on the friction surface and the increase in the thickness and strength of the adsorption membrane. Consequently, the lubrication performance increased slightly. An oil membrane with sufficient thickness was difficult to form on the high-temperature friction surface when the lubricating fluid had low viscosity. The oil membrane had the low bearing capacity and was easily damaged under loads. The lubrication in the friction pair was inadequate, and the friction coefficient increased accordingly.

The friction coefficients of the different lubricating fluids are exhibited in Fig. 9a. The addition of different surfactants to CNT nanofluids caused different friction coefficients, and the fluctuation in the friction coefficients significantly changed with time. Given the entire experiment, the friction coefficients of nanofluids with a non-ionic surfactant (APE-10 and OP-10) increased with time. The friction coefficient of nanofluids with OP-10 increased from 0.135 to 0.157 after 200 s because APE-10 and OP-10 exhibited high molecular mass and London force, thereby playing the dominant role in several dispersing mechanisms. Therefore, nanofluids showed a high dispersion stability in a static observation. The lubricating fluid was disturbed by the rotation sliding of the white alundum ball, and the stable system formed by London force, which was generated by an instantaneously induced dipole, was damaged throughout the frictional test, thus weakening the dispersion of CNTs and intensifying the aggregation of CNTs gradually. Consequently, a stable adsorption film was impossible to form, and the continuity of the adsorption film was destroyed; this result contradicted the lubrication performance.

The friction coefficient of nanofluids with a cationic surfactant (CTAB and TTAB) slightly decreased with time. The friction coefficients of nanofluids with CTAB decreased from 0.15 to 0.135, whereas those with TTAB decreased from 0.17 to 0.15. The reason for these results might be as follows: with the continued friction and wear process, the friction surface gradually smoothened, the wear changed from the violent wearing state to the stable wearing state gradually, and the



Fig. 9. (a) Friction coefficient, (b) Box plot of friction coefficient.

destroyed friction film was updated and supplemented timely, thereby contributing to the continuously satisfying lubrication performances [32]. The friction coefficient curves of nanofluids with anionic SDS and SDBS remained constant throughout the experiment, except for a slight fluctuation.

Fig. 9b illustrates that the average friction coefficient of the CNT nanofluids with APE-10 was the lowest (0.121), thereby indicating the optimal friction-reduction performance. The overall order of average friction coefficient was APE-10 (0.121) < CTAB (0.144) < OP-10 (0.148) < SDBS (0.156) < TTAB (0.160) < SDS (0.169). On the basis of the average friction coefficient, the comparative analysis was as follows:

- APE-10 versus OP-10. The friction coefficient of the CNT nanofluids added with APE-10 was the lowest, and the friction coefficients fluctuated slightly. APE-10 is a mixed surfactant, and the surface active molecules of APE-10 have high-efficiency synergism; in addition, APE-10 can adsorb onto the CNT-PPO interface, and a highefficiency synergistic effect exists between APE-10 and CNTs [54,55]. The specific performance might be as follows: different types of surfactants formed reversed micelles with different sizes and shapes, which filled different spatial spaces and formed stable dispersions. The nanofluids with OP-10 showed a favorable dispersion effect in the static observation. A possible reason was that the surfactant concentration reached the critical reversed micelle concentration. The other possible reason is that OP-10 molecules contain -O- and -OH polar groups, which will decrease the critical reversed micelle concentration of OP-10 sharply [45]. Therefore, a low concentration could form the reversed micelles and reach the saturated adsorption of molecular monolayer. The low concentration could change the internal and interface performances of the system significantly; that is, the OP-10 surfactant could develop high performances under a low concentration. The frictional test reported that the friction coefficient under nanofluids with OP-10 was not the lowest, and the disc surface quality analysis demonstrated the poor friction-reduction and antiwear performances, which were caused by the instability of the dominant London force in the frictional tests.
- *CTAB versus TTAB.* Fig. 9b displays that the average friction coefficient of nanofluids with CTAB was lower than that of nanofluids with TTAB. The possible reason was that the carbon chain of the lipophilic group of CTAB is longer than that of TTAB, and the distance among CNTs when CTAB was adsorbed onto CNTs is longer than that under CTAB. Thus, CNTs could uniformly disperse and easily form the uniform friction membrane on the friction pair

surface, thus contributing favorable friction-reduction and antiwear performances. Moreover, CTAB exhibited strong lipophilicity and low critical reversed micelle concentration in palm oil and performed efficiently under a low concentration, thereby causing it to have a high adsorption capacity on the CNT-PPO interface and improved adsorption stability. The experimental results of the static observation were opposite to the abovementioned analysis. Nanofluids with CTAB required a shorter time for layering deposition than those with TTAB, thus indicating a favorable dispersion effect of CNT nanofluids with TTAB. One possible reason is that TTAB possesses shorter hydrocarbon chain and stronger hydrophilicity of hydrophilic groups; thus, TTAB is apt to escape from oil and can easily be adsorbed onto the CNT surface, thereby contributing to the increase in adsorption capacity [45]. The other possible explanation was as follows: The carbon number on the straight alkane chain of the lipophilic group exceeds 16, and the static observation time is longer than that of the frictional test, thus causing the curling up of the long chain; the cross-sectional area of single surfactant molecules was further increased [47]. Ultimately, the saturated adsorption capacity of surfactant and the loosening of the adsorption layer were decreased.

• *SDBS versus SDS.* Nanofluids with SDBS are superior to those with SDS in terms of the average friction coefficient and static dispersion performance; the possible reasons are that SDS has a large HLB value that leads to the poor oil solubility, and the solubility of SDS powder in palm oil is relatively small, thereby resulting in a small number of micelles formed by SDS molecules; consequently, the dispersion effect is the poorest [53]. Macroscopically, the experimental results of a static observation showed that SDS is layered up in the lubricating fluid, and abundant SDS is no longer dissolved in the lubricating fluid and cannot effectively disperse.

Certain information is displayed in Fig. 9b. First, the friction coefficients of nanofluids with nonpolar ionic surfactant OP-10 and cationic surfactants CTAB and TTAB varied extensively. The extensive variation in the friction coefficient of nanofluids with OP-10, as presented in Fig. 9a, was due to the friction coefficient curve steadily increased after 200 s, accompanied by a small fluctuation. Second, the friction coefficients of nanofluids with cationic surfactants CTAB and TTAB fluctuated significantly throughout the experiment, and the maximum friction coefficient influenced the mean considerably, thereby indicating a mutual corroboration as illustrated in Fig. 9a. The violent fluctuation might be the consequence of the accumulation and exfoliative discharge of cutting debris.

4.4. Effects of surfactants on the antiwear performance of CNT nanofluids

4.4.1. Roughness

The surface roughness value of the disc samples was measured after the frictional test to discuss the frictional wear of samples. Roughness measurement and mean were gained from 3 points to 5 points of each disc sample, and the point that is closest to the roughness mean was selected for evaluation. The roughness values of disc samples under different nanofluids are depicted in Fig. 10, and the roughness profile curves of disc samples under different CNT nanofluids are demonstrated in Fig. 11. The roughness value order under different lubrication conditions was APE-10 (0.48 um) < OP-10 (0.518 um) < SDBS $(0.552 \, \text{um}) < \text{CTAB}$ $(0.533 \, \text{um}) < \text{TTAB}$ $(0.691 \, \text{um}) < \text{SDS}$ (0.839 µm). The minimum roughness value was achieved by CNT nanofluids with APE-10; therefore, they achieved the optimal lubrication and tribological properties. Scratches were only one part of the discsampling section for roughness measurement, and the roughness value could not comprehensively reflect the surface quality and lubrication performance of nanofluids.

4.4.2. Maximum scratch depth

MATLAB program was used to generate the scratch profile curves based on the original roughness data for fully reflecting the grinding performance of white alundum balls and the antiwear performances of different nanofluids with surfactants. Fig. 11 displays that the maximum scratch depth order under different nanofluids was APE-10 $(7.024 \,\mu m) < TTAB$ (7.790 µm) < SDBS $(8.469 \,\mu m) < OP-10$ $(9.238 \,\mu m) < CTAB (9.608 \,\mu m) < SDS (10.057 \,\mu m)$. The nanofluids with APE-10 contributed the optimal antiwear performance. The dispersion effect of CNTs under CNT nanofluids with APE-10 was stable, and CNTs easily formed a stable friction film, thereby leading to the reduction in surface roughness. A low surface roughness enhanced the capacity of film forming and the thickness of the oil film. A virtuous circle was formed in this way. The optimal antiwear performance was achieved under nanofluids with APE-10.

4.4.3. Material removal rate

Material removal rate is the material volume on the disc removed by the white alundum ball per unit time. The antiwear performance of different CNT nanofluids can further be analyzed by material removal rate. Area ratio (η) can reflect the material removal rate, and η is negatively correlated with the material removal rate. The related formula of area ratio is expressed as follows:

$$\eta = (S_1 + S_2)/S_3 \tag{10}$$

where η is the area ratio, $S_1 (\mu m^2)$ and $S_2 (\mu m^2)$ are the cross-sectional areas of plastic accumulation caused by disc extrusion by the white alundum ball, and $S_3 (\mu m^2)$ is the cross-sectional area of the furrows produced by the white alundum ball cutting of the disc. The cross section of a scratch is illustrated in Fig. 12. The values of S_1 , S_2 , and S_3 were calculated by MATLAB software. In Fig. 13, the order of η of the six disc groups in terms of material removal rate is TTAB < SDBS < SDS < APE-10 < OP-10 < CTAB. Therefore, the order of nanofluids in the antiwear performance is CTAB > OP-10 > APE-10 > SDS > SDBS > TTAB. Fig. 14 demonstrates that the disc scratches under nanofluids with OP-10 presented obvious narrow gully and high maximum scratch depti; this result obviously disagreed with the white alundum ball profile. This result might be due to the large or several cutting debris piled up in the grinding zone to cut the disc as the "third body" under the extrusion of white alundum balls [56].

4.4.4. SEM morphology

Disc surface was observed by SEM to disclose the lubrication mechanism of different CNT nanofluids. The SEM morphologies under six types of CNT nanofluids are depicted in Fig. 14. The worn surfaces under nanofluids with OP-10, APE-10, and TTAB were relatively smooth, without evident furrows and scratches. Particularly, the worn surfaces under nanofluids with APE-10 and TTAB were relatively rough. The worn surface under nanofluids with OP-10 hardly had large furrows and wrinkle superposition (Fig. 14c). The worn surface under nanofluids with OP-10 was relatively smooth, but the maximum scratch depth was 9.238 µm, as exhibited in Fig. 11. This result might be due to the relatively smooth worn surfaces were against the high storage capacity of nanofluids, which caused it to be impossible to generate friction film. Evident scratches and furrows were observed from the worn surfaces under nanofluids with CTAB, SDBS, and SDS, accompanied with large-sized nonseparated cuttings at the furrow edges (Fig. 14b, d, and 14e). The possible reason might be that the aggregated CNTs and adhered chip were ground into blocks given the high nanofluid viscosity, which would cut plow the disc as the "third body" under the extrusion of white alundum balls [56]. However, with limited hardness and cutting capability, the nonseparated chip could not be cut off completely and effectively from the disc. Fig. 14c and f depict that obvious accumulation of CNTs existed on the friction surface. The CNT aggregations were extruded by a white alundum ball in the cutting zone and then accumulated on the friction surface.

(a): PPO + 2 wt% (CNTs) + 3 wt% (APE-10); (b): PPO + 2 wt% (CNTs) + 3 wt% (CTAB); (c): PPO + 2 wt% (CNTs) + 3 wt% (OP-10); (d): PPO + 2 wt% (CNTs) + 3 wt% (SDS); (e): PPO + 2 wt% (CNTs) + 3 wt% (SDS); (f): PPO + 2 wt% (CNTs) + 3 wt% (TTAB).

5. Conclusions

This study evaluated the dispersing properties of different types of surfactants. The molecular structures and physicochemical properties of different surfactants were analyzed, and their dispersing mechanisms were studied. Six surfactants were selected for experimental verification. CNT nanofluids added with different surfactants were observed under static conditions. A contrast analysis on the viscosity and friction coefficient of the lubrication of different CNT nanofluids and the wearing loss of discs was performed to compare and verify the dispersion mechanism. On the basis of the experimental results, the following conclusions were drawn:

- (1). Among the six surfactants, the CNT nanofluids with APE-10 obtained the longest time without deposition (> 45 h) and the highest viscosity (345.7 MPa s), thereby demonstrating the most outstanding dispersion stability.
- (2). Further investigation showed that the CNT nanofluids with APE-10 gained the lowest friction coefficient (0.121), lowest maximum scratch depth ($7.024 \,\mu$ m), lowest roughness value ($0.48 \,\mu$ m), second lowest material removal rate, and favorable frictional



Fig. 10. Roughness value of CNT nanofluids with different surfactants.



Fig. 11. Roughness profile curve. (a): PPO+2 wt% (CNTs)+3 wt% (APE-10); (b): PPO+2 wt% (CNTs)+3 wt% (CTAB); (c): PPO+2 wt% (CNTs)+3 wt% (OP-10); (d): PPO+2 wt% (CNTs)+3 wt% (SDBS); (e):PPO+2 wt% (CNTs) + 3 wt% (SDS); (f): PPO + 2 wt% (CNTs) + 3 wt% (TTAB).



Fig. 12. Cross section of a scratch.





Fig. 14. SEM morphology of the worn surface.

surface morphology, thereby indicating the excellent tribological property, antiwear, and friction-reduction performance.

- (3). The order of viscosity and the average friction coefficient of nanofluids with different surfactants were strongly correlated. The high viscosity of nanofluids implied a stable oil film, thus exerting a favorable friction-reduction effect.
- (4). The static observation results and experimental evaluation presented that APE-10 was optimal to be added to CNT nanofluids as surfactants, which indicated that the "synergism" of a mixed surfactant exerted excellent dispersion effect and tribological property.

Acknowledgments

This research was financially supported by the National Natural Science Foundation of China (51575290), the Major Research Project of Shandong Province (2017GGX30135 and 2018GGX103044), and the Shandong Provincial Natural Science Foundation of China (ZR2017PEE002 and ZR2017PEE011.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https:// doi.org/10.1016/j.triboint.2018.10.025.

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