



## A novel approach to use some fatty acid based ionic liquids and reduced graphene oxide as lubricant additives

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### ARTICLE INFO

Received: 04/03/2025

Accepted: 30/03/2025

Published: 30/03/2025

#### Keywords:

Graphene;  
lubricant additive;  
Ionic liquids;  
waste vegetable oil;  
fatty acids;  
triethyl amine; aliquat;  
octyl amine,  
1-methyl imidazole.

### ABSTRACT

Reduced graphene oxide (rGO) has many advanced characteristics such as thermal and electric conductivity, layered structure and its layers can easy separate when sliding on each other. Therefore, it is very suitable for using as a lubricant additive. In this work rGO and eight ionic liquids triethyl ammonium oleate ([TEA][Oleate]), octyl ammonium oleate ([Oca][Oleate]), trialkyl methyl ammonium oleate (Alkyl: C<sub>8</sub>H<sub>17</sub> and C<sub>10</sub>H<sub>23</sub>) ([Aliquat][Oleate]), octyl methyl imidazolium oleate ([OMIM][Oleate]), octyl methyl imidazolium stearate ([OMIM][Stearate]), octyl methyl imidazolium stearate ([OMIM][Stearate]), Octyl methyl imidazolium carboxylate (fatty acid separated from coconut oil) ([OMIM][CO]), Octyl methyl imidazolium carboxylate (fatty acid separated from rubber seed oil) [OMIM][RSO]), Octyl methyl imidazolium carboxylate (fatty acid separated from waste oil) ([OMIM][WO]) with different cationic and anionic structures were used as friction-reducing additives for lubricants. The role of ionic liquids in stabilization rGO in lubricants was investigated. In addition, influence of ionic liquid structures on stabilization of rGO in lubricants was also evaluated. The friction moment was reduced by 73%-63% compared to 58% when rGO was added. Among ionic liquids with the same anion [Oleate], the ionic liquid from cation [OMIM] led to the most stable additive system in lubricant and the highest friction-reducing ability, followed by [Oca], [Aliquat] and [TEA]. Among ionic liquids with the same cation [OMIM], the stability and friction-reducing ability of ionic liquids followed the order of anion: [Oleate] > [RSO] > [WO] > [Stearate] > [CO].

### Introduction

Graphene, a two-dimensional hexagonal lattice structure of carbon, has been recognized as the most outstanding material of this decade with a wide range of applications. Graphene has high mechanical strength, weak van der Waals interaction between atomically thick layers, so these layers are easily

separated under force, resulting in low deformation; moreover, when separated, the layers have very smooth surfaces. In addition, graphene is chemically inert, has high thermal conductivity and a large surface area. Graphene has an ultra-thin structure even when it has several layers, it can be used as a lubricant for systems with nano- or micro-sized vibrating, rotating and sliding parts to reduce

adhesion, friction and wear. Due to all these properties, graphene is widely used as a lubricant and lubricant additive to produce low friction and wear. Many studies have used graphene as a solid lubricant, as an additive to lubricants and have shown very promising results [1,2]. Studies have shown that few-layer graphene [3-6] can reduce wear by up to 104 times and friction by 105-106 times. In addition, it has been shown that graphene works equally well in wet and dry environments, while conventional solid lubricants such as ordered pyrolytic graphite do not have this property. However, in a lubricant environment, graphene is prone to agglomeration into large particles, reducing the surface area and lubrication efficiency, so it is important to study their stable dispersion. Many graphene modification methods have been used to stabilize and disperse graphene in oil. Li *et al.* [7] fabricated graphene with high specific surface area by thermal reduction of graphene oxide and stabilized this graphene in poly( $\alpha$ -olefin) (PAO-6) lubricant for up to 4 days. When 0.5 wt % graphene was added to the PAO-6 lubricant, the coefficient of friction and wear depth were reduced by 44% and 90%, respectively.

Dou *et al.* fabricated graphene wrinkled spheres like micro-balls that can create a ball bearing effect, changing direction during sliding, which prevents two contact surfaces from being "locked" together when sliding on each other [8] to serve as an effective lubricant additive in PAO-4. The graphene wrinkled spheres were more stable in the base oil than graphite, carbon black, and commercial reduced graphene oxide.

Liu *et al.* were the first scientist who use ionic liquids as lubricants [9] and reported that the addition of a small amount of water ( $\leq 5$  wt%) to ILs could improve the anti-wear properties of some metal-metal interfaces [10,11]. The lubrication mechanism of ILs was explained by surface smoothing after mechanical abrasion, the formation of films on the surface, and the creation of an electrical double layer of IL molecules, which increased the local viscosity near the surface and the load-carrying capacity, and reduced friction and wear.

Recently, José Sanes *et al.* discovered a synergistic effect when using the ionic liquid 1-octyl-3-methylimidazolium tetrafluoroborate and graphene as lubricant additives simultaneously [12]. Graphene, ionic liquids and their combinations were studied as additives to isoparaffinic base oils without additives,

under relatively low load conditions at room temperature and as additives to high-performance finished oils under more severe conditions, at 150°C. In the case of isoparaffinic base oils, a synergistic effect between graphene and ionic liquids was observed.

In this work, graphene was prepared by oxidation of graphite to graphene oxide (GO), which has oxygen functional groups, and then reduction of GO to graphene or reduced graphene oxide (rGO) having no or very low number of oxygen functional groups. Obtained rGO and 8 fatty acid based ionic liquids with different structure were used as additives for lubricants. Four popular cation such as triethyl ammonium, octyl ammonium, aliquat, octyl methyl imidazolium were used to find the influence of cation structure on the friction properties of IL. Fatty acids based ionic liquids were chosen because they are high compatibility with lubricants deriving from vegetable oils. The stability of rGO and friction moment reduction was investigated to find the role of ionic liquid in increasing the lubricant performance of rGO.

## Experimental

### Materials

Zinc acetate dihydrate 99%, iron (III) nitrate nonahydrate 99%, hydrogen peroxide 30%, hydrochloric acid 35%, sulfuric acid 98%, sodium hydroxide 99%, ethanol 99%, potassium permanganate 99%, methylene blue, acetic acid 99% and acetone 99% were supplied by Xilong Company; ascorbic acid 99% (Fisher); graphite (Merck); ammonia solution 25% (GHTech), Rhodamine B (Oxford Lab Fine Chem LLP) are used without further purification. 8 ionic liquids: [TEA][Oletate], [Oca][Oletate], [Aliquat][Oletate], [OMIM][Oleate], [OMIM][Stearate], [OMIM][Stearate], [OMIM][CO], [OMIM][RSO], [OMIM][WO] are synthesized and provided by the laboratory of the Hanoi University of Mining and Geology [13,14].

### Preparation of rGO

#### Preparation of GO

The procedure follows the method of Bui *et al.* [15,16] and the details as follows. 2.5g NaNO<sub>3</sub> and 5g graphite were slowly added to 115mL H<sub>2</sub>SO<sub>4</sub> 98% solution kept at 3-5°C. The stirring with a magnetic stirrer was continued for 30 min and then 15g KMnO<sub>4</sub> was gradually added while keeping temperature of the below 15°C. The temperature of the reaction mixture was increased to 35°C and kept stirring for about 30

min; then, distilled water was gradually introduced to the reaction mixture to keep the temperature at around 45°C. Then temperature was increased to 95°C and the reaction mixture was stirred for extra 15 min at this temperature. Next, reduce the temperature of the system slowly to room temperature, add 50mL H<sub>2</sub>O<sub>2</sub> solution to the mixture and stir for 20 minutes. The solid of final mixture was centrifuged with 8000 rpm in 6 min to get GO. Then, the GO were dispersed in HCl 0.1M solution, stirred for 15 min and centrifuged for purifying GO from impurities part. After drying at 70°C - 80°C, grinding with a ceramic mortar, the resulting product is GO.

#### Reduction of GO to rGO

The procedure follows the method of Bui *et al.* [15,16] and the details as follows. 10 g of ascorbic acid was dissolved in 100 ml of distilled water. 1 gram of GO was dispersed in 100 ml of distilled water by stirring and ultrasonic for one hour to form a suspension. Then 3 grams of gelatine was gradually introduced, the temperature was increased to 70°C. 100 ml aqueous solution containing 10 g ascorbic acid was added slowly in the suspension in 2 hours. Then mixture was stirred at 70°C for 12 hours. The resulting mixture was centrifuged and filtered, washed with water and ethanol, and dried at 60°C. The obtained product was reduced graphene oxide (rGO).

#### Stability evaluation of rGO and ionic liquid mixtures in lubricants.

Reduced graphene oxide (rGO) and ionic liquid [TEA][Oleate] was mixed to form a dispersion of 0.20 wt% rGO in IL, then this dispersion was added up to 2 wt% to lubricant to form a mixture containing 0.002 wt% rGO and 2 wt% IL denoted as the rGO/[TEA][Oleate] 0.2%/CBT 2% mixture. The mixture was shaken, stirred and ultrasonic to obtain a homogeneous system. The procedure was repeated with the remaining 7 ionic liquids listed in Section 2.1 to get 8 samples (rGO + IL) 0.2% (Table 1).

Table 1. List of ionic liquids and samples in this research

Ionic liquids	Abbreviation
[TEA][Oleate]	rGO/[TEA][Oleate] 0.2%/CBT 2%
[Oca][Oleate]	rGO/[Oca][Oleate] 0.2%/ CBT 2%
[Aliquat][Oleate]	rGO/[Aliquat][Oleate]0.2%/CBT 2%
[OMIM][Oleate]	rGO/[OMIM][Oleate] 0.2%/ CBT 2%
[OMIM][Stearate]	rGO-OA/[OMIM][Stearate] 0.2%/ CBT 2%
[OMIM][CO]	rGO-OA/[OMIM][CO]0.2%/CBT 2%
[OMIM][RSO]	rGO-OA/[TEA][WO] 0.2%/ CBT 2%
[OMIM][WO]	rGO-OA/[Aliquat][RSO]0.2%/CBT 2%

<https://doi.org/10.62239/jca.2025.014>

The UV-VIS of dispersions was measured after mixing at different time intervals. From the UV-VIS measurement results, the relative concentration of solids can be calculated according to formula (1). Based on this relative concentration, the stability of solids in liquids can be assessed:

$$\text{Relative con.} = \frac{\text{Absorption at certain time}}{\text{Absorption at ininitial time}} * 100\% \quad (1)$$

#### Evaluation of the friction moment reduce ability moment for lubricants used in drilling fluids

The friction performance of lubricants in drilling fluids is determined (on the EP/Lubricity Tester) according to the technical standard PĐ VSP-000-PK-650 of the Vietnam-Russia Oil and Gas Joint Venture. The drilling fluid used as a basis for measuring lubrication has a specific gravity of 1.40-1.45 G/cm<sup>3</sup>; pH 9-9.5 and has components and rheological parameters according to the technical standard PĐ VSP-000-PK-637 of the VietsoPetro. The proposed formulation of drilling fluids is a water-based inhibitor solution system.

The friction reduction indicates the lubricity of lubricants in drilling fluids and is calculated according to the following formula:

$$\text{Friction moment reduction} = \frac{(f_1 - f_2) * 100}{f_1} \%$$

Where:  $f_1$  readings on the meter without adding lubricant additives;  $f_2$  readings on the meter with adding lubricant additives.

The friction reduction was measured at room temperature and after heating (at 130°C during 16 h) for evaluate the stability of the lubricant containing additives.

## Results and discussion

#### Stability of rGO in ionic liquid and lubricant mixtures

In this section, the stability of the samples (rGO + IL) 0.2% was evaluated. UV-VIS spectra were measured immediately after mixing and after time intervals to evaluate the stability (settling of solids) to a rGO in lubricant sample with an equivalent concentration but without ionic liquid. The results are presented in Table 2.

It can be seen that when GO was reduced to rGO, the stability of rGO is higher, this is due to the oxygen-containing functional groups of GO were reduced during reducing GO to rGO. In the absence of IL, the concentration of GO in lubricant decreases quite rapidly over time, after 20 days the relative concentration is only 69.7%.

Table 2. Relative concentration changes over time of GO and rGO samples dispersed in IL-free lubricant

Time (day)	0	5	10	15	20
Relative concentration of GO (%)	100	70.1	64.8	62.7	62.2
Relative concentration of rGO (%)	100	77.6	72.3	70.2	69.7

### *Influence of cationic structure of IL on the stability of rGO in IL/lubricant mixtures*

In this section, 4 ionic liquids used were prepared from 4 cations triethyl ammonium, octyl ammonium, aliquat, octyl methyl imidazolium and oleate anion, other 4 ionic liquids and from cation octyl methyl imidazolium and 4 anions stearate, carboxylate of fatty acids of coconut oil, rubber seed oil, waste oil. The influence of the cationic structure on the stability of rGO and ILs in lubricant was evaluated and results are shown in Figure 1.

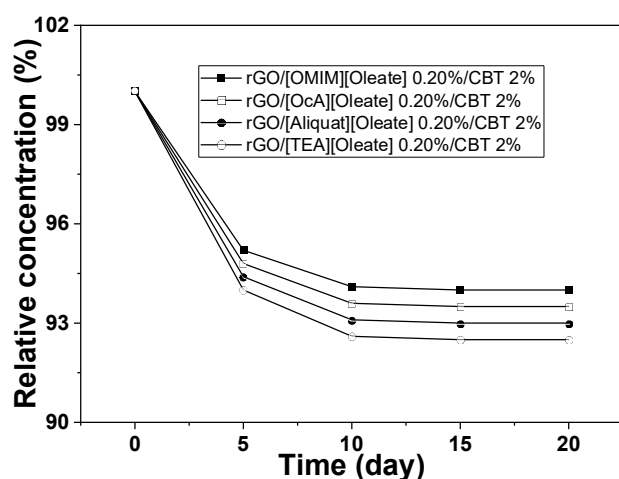


Figure 1: Influence of cationic structure of ionic liquid on the stability of rGO in IL and lubricants: GO concentration in IL is 0.20%; IL concentration in lubricant is 2%.

It can be seen from the results in Figure 1 that in all samples, there was a decrease in rGO concentration over time but not much. For example, after 5 days, the relative concentration of rGO in [OMIM] IL mixtures decreased from about 100% to 95%-95.4%; in [OcA] IL mixtures decreased from 100% to 94.6%-95%; in [Aliquat] IL mixtures decreased from 100% to 95.2%-94.6%; in [TEA] IL mixtures decreased from 100% to 93.8%-94.2%;

After 20 days, the relative concentration of rGO in [OMIM] IL mixtures decreased from about 100% to 93.7%-94.2%; in [OcA] IL mixtures decreased from

100% to 93.2%-93.7%; in [Aliquat] IL mixtures decreased from 100% to 92.7%-93.2%; in [TEA] IL mixtures decreased from 100% to 92.2%-92.7%; Therefore, it can be seen that the rGO is most stable in the [OMIM] based ionic liquid mixtures, then in the [OcA] based IL mixtures, in [Aliquat] based IL mixtures and is the least stable in the [TEA] based IL mixtures, specifically, in the following order:

$$[\text{OMIM}] > [\text{OcA}] > [\text{Aliquat}] > [\text{TEA}] \quad (2)$$

The rGO contains many polar functional groups, therefore, it easily interacts with the cations of the ionic liquids. Specifically, the [OMIM] cations with  $\text{C}_8$  chains are easier to interact with rGO than [OcA], while [Aliquat] is more difficult to interact with rGO because it has 3 long alkyl chains surrounding the positively charged nitrogen atom.

Furthermore, aliquat has four electron-donating alkyl groups that reduce the positive charge on the nitrogen atom, which also leads to a decrease in interaction with rGO. The [TEA] cation is an ammonium salt with three electron-donating ethyl groups and a relatively short chain that facilitates rGO access to the nitrogen atom, however, the ionic liquids formed from this anion have quite high viscosity and melting point, which reduces the dispersion of rGO.

### *Influence of anionic structure of ILs on the stability of rGO system in mixture of ionic liquids and lubricants*

In this section, the ionic liquids used to mix with rGO are made up of cation octyl methyl imidazolium and 5 anions: oleate, stearate, fatty acid carboxylate of coconut oil, rubber seed oil, waste oil. The influence of cation structure on the stability of rGO in ILs is evaluated and shown in Figure 2. It can be seen from the results in Figure 2 that in all samples rGO concentration in mixture decreased over time. For example, after 5 days and 20 days, the relative concentration of rGO in [Oleate] IL mixtures decreased from approximately 100% (0 days) to 94%-95.2% and 92.5%-94%, respectively; in [RSO] IL mixtures decreased from 100% to 94.2%-95.4% and 92.7%-94%; in [WO] IL mixtures decreased from 100% to 94.1%-95.3% and 92.6%-94.1%; in [Stearate] IL mixtures decreased from 100% to 93.8%-95% and 92.2%-93.7%; in [CO] IL mixtures decreased from 100% to 93.9%-95.1% and 92.3%-93.8%. It can be concluded that the rGO disperses stably in the group [Oleate] based ionic liquids, then [RSO], [WO], [Stearate] based ILs disperses least stably in based on [CO] IL mixture, following the order:

$$[\text{Oleate}] > [\text{RSO}] > [\text{WO}] > [\text{Stearate}] > [\text{CO}] \quad (3)$$

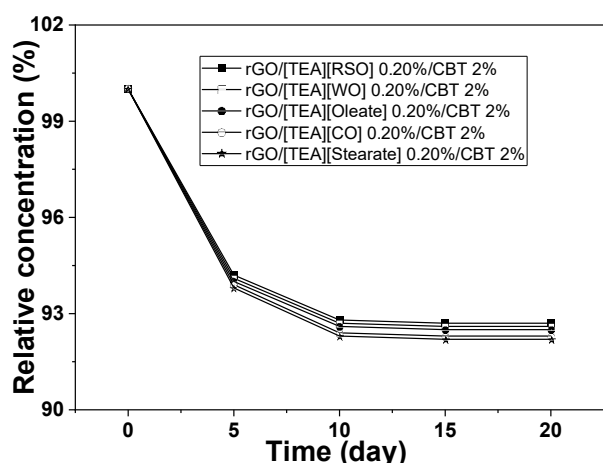


Figure 2: Influence of anionic structure of ionic liquid on the stability of rGO in IL and lubricant mixture, rGO concentration in IL is 0.20%, ionic liquid concentration in lubricant is 2%.

rGO contains many oxygen-containing functional groups, therefore, it easily disperses in polar ionic liquids. Ionic liquids based on unsaturated fatty acid anion are more polar and contains high electron density  $\pi$  bonds, so that it is easily compatible with rGO which has polar O-H groups. Because content of unsaturated bonds in ionic liquids based on oleic acid, fatty acids of rubber seed oil and waste oils decreased, then the dispersion of rGO in ionic liquids follows the order (3). Coconut oil fatty acids are mainly saturated acids, so that the dispersion of rGO in [CO] ionic liquids reduced. Similarly, the dispersion of rGO in stearate ionic liquids is the lowest.

#### Friction reduction test results of rGO and ionic liquid mixture

8 mixtures containing ionic liquid and rGO were used in the friction reduction test (Table 3). The results of Table 3 show that the friction moment of the additive-containing lubricants varies depending on the structure of the ionic liquids. In addition, for ionic liquids with the same cation, the friction moment reduction varies according to the nature of the anion and vice versa.

#### Friction performance of lubricants before calcination

##### Influence of ionic liquid cation structure

With the same anion [oleate], the friction moment of the additive (rGO and ionic liquids) containing lubricant decreased most when using ionic liquids from the cation [OMIM] (73%), followed by [OcA] (72%), [Aliquat] (70%) and finally [TEA] (71%). It can be seen that the change in friction moment is closely correlated with the stability of rGO in the lubricant. rGO stable most in the ionic liquid containing [OMIM], followed by

the cation [OcA], the cation [Aliquat] and finally cation [TEA]. When IL and rGO are mixed with the similar lubricant, the order was observed (Table 3).

Table 3. Friction reduction results of additive samples containing rGO and ionic liquids

Abbreviation	Reduction in friction moment of lubricant containing rGO and ionic liquid (%)	
	Before heating	After heating*
GO	55	50
rGO	58	54
rGO/[TEA][Oleate] 0.2%	73	65
rGO/[OcA][Oleate] 0.2%	72	65
rGO/[Aliquat][Oleate] 0.2%	71	63
rGO/[OMIM][Oleate] 0.2%	70	62
rGO/[OMIM][Stearate] 0.2%	70	55
rGO/[OMIM][CO] 0.2%	69	61
rGO/[OMIM][RSO] 0.2%	67	54
rGO/[OMIM][WO] 0.2%	63	50

\* at 130°C during 16 h

#### Influence of anion structure of ionic liquids

With the same cation [OMIM], the friction moment of the additive (rGO blend with ionic liquids) containing lubricant decreased most when using the ionic liquid from the anion [RSO] (73%) and the anion [WO] (73%), then the anion [Oleate] (71%), and finally the anion [CO] (70%) and [Stearate] (70%). Therefore, it can also be seen that the change in friction moment is closely correlated with the stability of rGO in the lubricant. When the ILs and rGO were mixed into the lubricant, the order of stability of rGO was similar. The data in Table 3 also shows that when using IL together with rGO, the frictional moment is reduced more than when using rGO alone.

#### Friction performance of lubricants after calcination

Similar to before calcination, the friction moment reduction of lubricants after calcination at 130°C (for 16 hours, with pH increase = 8.5 – 8.7) also depends on the structure of the cation and anion of the ionic liquids.

With the same cation [OMIM], the friction moment of the lubricant containing rGO-ILs decreased most with the ionic liquids from anion [RSO] (65%) and [WO] (65%), then [Oleate] (63%), and finally [CO] (62%) and [Stearate] (62%). This order of results is similar to the frictional moment measurements before calcination.

With the same anion [Oleate], the friction moment of the lubricant containing rGO and ionic liquids decreased the most with ionic liquid from the cation [OMIM] (65%), then [Aliquat] (65%), [Oca] (63%) and finally [TEA] (62%). It can be seen that the change in friction moment is closely correlated with not only the stability of rGO but also the stability of the ionic liquids in the lubricant. rGO is most stable in the ionic liquid group of ionic liquid from [OMIM] followed by the [Oca], the [Aliquat] and finally [TEA]. However, the reduction in friction moment the lubricant with the ionic liquid from [Oca] and [TEA] decreased significantly compared to before calcination, but was still higher than the standard (>50%). This can be explained by the fact that these ionic liquids are not stable at high temperatures, therefore, the ability to disperse rGO is reduced leading to reduced lubrication ability.

## Conclusion

The stability of rGO in lubricants was determined. When GO was reduced to rGO, the dispersion ability in lubricants increased. This is due to when GO was reduced the number of oxygen functional groups decreased made rGO more compatible with the alkyl groups of hydrocarbons of the lubricant.

When ionic liquids were added to the mixture rGO and lubricant, the stability of the rGO in the lubricant increased significantly. rGO the most stable in the [OMIM] based ionic liquid mixtures, followed by the [Oca] based IL, [Aliquat] and the [TEA] based IL mixtures being the least stable, specifically, followed the order: [OMIM] > [Oca] > [Aliquat] > [TEA]. The rGO was most stable in the [Oleate] based ionic liquid mixtures, followed by the [RSO] based IL and least stable in [WO], [Stearate] and the [CO] based ILs, specifically, followed the order: [Oleate] > [RSO] > [WO] > [Stearate] > [CO].

The higher stability of rGO in lubricants than of GO is explained by the decrease in oxygen functional groups of rGO which increase the compatibility of rGO with the alkyl groups of hydrocarbons of the lubricant

The friction moment reduction of lubricant containing GO and ionic liquids depended on the structure of the cation and anion of the ionic liquid. This closely correlated with the stability of the rGO-IL in the lubricant. Friction moment reduction reached 69%-73% before calcination and 50%-65% after calcination. The results show that fatty acid based ionic liquids and reduced graphene oxide are potential candidate as lubricant additives.

## Acknowledgments.

This work was supported by the Ministry of Industry and Trade, Viet Nam [ĐT.CNKK.QG.002/23]

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