Effect of saline water on the synergistic interaction between diesel and Triton X-100 in the flotation of oxidized coal

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Abstract: In this study, the effect of medium saline water on the synergistic interaction between diesel and Triton X-100 in the flotation of oxidized coal was investigated. The results showed that the flotation yield of oxidized coal in saline water was higher than that in de-ionized (*DI*) water due to the promotion of diesel adsorption, which was attributed to the screening of electrostatic repulsion between diesel droplets and coal particles in saline water. Meanwhile, the flotation of oxidized coal could be significantly improved when Triton X-100 was added with diesel as a composite collector, and less Triton X-100 was required in saline water than that in *DI* water to achieve the same true flotation yield, indicating that saline water could increase the effectiveness of Triton X-100 in improving oxidized coal flotation. The mechanism study revealed that Triton X-100 was able to promote diesel adsorption on oxidized coal through emulsification, thus increasing the surface hydrophobicity of oxidized coal through hydrogen bonding between the headgroups of Triton X-100 and the oxygenated groups on coal surfaces. The non-ionic characteristic of Triton X-100 ensured its capability of enhancing oxidized coal flotation in both *DI* water and saline water.

Keywords: saline water, surfactants, adsorption, flotation, coal oxidation

1. Introduction

Diesel is often used as the collector of coal flotation to enhance coal surface hydrophobicity and promote particle-to-bubble attachment. Normally, diesel is ready to adsorb on the naturally hydrophobic coal surface (Laskowski, 2001). However, coal oxidation makes coal surface hydrophilic and prevents diesel adsorption. As a result, coal flotation deteriorates significantly with increasing degrees of coal surface oxidation (Sun, 1954; Sarikaya, 1995; Chang et al., 2017; Xing et al., 2017). The poor floatability of oxidized coal renders a large amount of coal particles to be wasted in flotation tailings, which has been a big problem faced by most coal preparation plants. In the previous work (Chang et al., 2017), X-ray photoelectron spectroscopy (*XPS*) was used to analyse the surface chemistry of oxidized coal, and the atomic percentage of oxidized carbon (at.%) was developed as an index to quantify coal surface oxidation. It was found from this study that the true flotation of oxidized coal became impossible when coal oxidation degree exceeded a critical value if flotation reagents remain unchanged.

The amphiphilic surfactants composed of polar groups and nonpolar hydrocarbon chains have often been used as polar collectors to improve the flotation of oxidized coal (Harris et al., 1995; Renhe Jia 2000; Dey, 2012; Qu et al., 2015; Gui et al., 2017; Wang et al., 2018). It is recognized that surfactants can adsorb on the oxidized coal through interaction between the head groups of surfactants and oxygen-containing groups on coal surfaces with surfactant tails orienting outwards, which is called "head-on" adsorption, thus increasing the hydrophobicity of oxidized coal. Renhe Jia (2000) found that the non-ionic THF ester surfactants were more effective than an oily collector when floating both oxidized and non-oxidized

coal, and the dosage of non-ionic surfactant required was much less than oily collector to achieve the same combustible recovery. Cebeci (2002) reported that in the flotation of lignite coal, the adsorption of kerosene on coal surface was enhanced by emulsifier and surfactant, and coal flotation performance was significantly improved. In previous work (Chang et al., 2019), it was found that composite collector consisting of nonpolar collector and polar collector was able to improve oxidized coal flotation with polar collector targeting oxidized coal surface area and nonpolar collector targeting un-oxidized coal surface area.

All previous findings were achieved in de-ionized (*DI*) water, which is different from saline water since flotation is sensitive to water quality. In Australia, Chile, South Africa, and many other countries, due to the scarcity of *DI* water and stringent regulations on the discharge of industrial water, water with high ionic strength, such as recycled water, underground water, and seawater, has often been used by mineral processing plants, particularly in froth flotation. In literature, it is documented that salts can significantly influence the flotation behaviour of coal particles. Several studies reported that the flotation of hydrophobic coal can be improved in saline water (Yoon, 1982; Yoon and Sabey, 1982; Kurniawan et al., 2011; Liu et al., 2013; Wang and Peng, 2014). Besides, our recent study revealed that coal flotation in saline water had better tolerance to coal surface oxidation than that in *DI* water due to the screening of electrostatic repulsion between coal particles and air bubbles, the generation of smaller bubbles, and higher froth stability (Chang et al., 2018). Nevertheless, when diesel was added with surfactants, the effect of saline water on the synergistic interaction between diesel and surfactants has not been reported.

In coal flotation, inorganic ions in saline water may influence the synergistic interaction between oil collector and surfactants either by affecting surfactant adsorption or oil emulsification. A comprehensive review about the influence of electrolytes on surfactant adsorption at the solid-liquid interface in flotation has been published elsewhere (Chang et al., 2018). In general, the electrolytes can significantly influence surfactant adsorption on minerals by affecting the solubility and aggregation of surfactants (Yu et al., 2012; Ahmadi et al., 2014), by affecting the adsorption density of surfactants (Nevskaia et al., 1996; Postmus et al., 2008; Xing and Rankin, 2013; Zhang et al., 2015), by modifying the morphology and structure of adsorbed surfactants (Domínguez, 2009; Liu et al., 2014). Furthermore, the presence of electrolytes may also influence the emulsification of oil. It was reported by Mei et al. (2011) that electrolytes could significantly increase the coalescence of emulsion droplets by screening the electrostatic repulsion among them. Overall, the effect of electrolytes on surfactant adsorption and oil emulsification is closely related to the valance and concentration of ions, which may vary from each other. Since the adsorption of ionic surfactants on mineral surfaces was easily affected by electrolytes (Chang et al., 2018), in this work, the non-ionic surfactant Triton X-100 was selected as the polar collector for oxidized coal flotation to avoid the interferences from salts.

In this study, synthetic saline water was prepared to simulate the typical process water used in coal preparation plants. The effect of saline water on the synergistic interaction between diesel and Triton X-100 in the flotation of oxidized coal was studied in detail.

2. Materials and methods

2.1. Materials

The coal sample used in this study was bituminous coal obtained from Bulli Mine, New South Wales, Australia. The combustible matter content, ash content, and moisture content of this coal sample was 89.95 wt.%, 7.93 wt.%, and 2.12 wt.%, respectively. In this work, the coal sample was crushed and ground to 80 wt.% below 200 μm to be consistent with the particle size used in most coal preparation plants. Coal sample with 13.20 at.% surface oxidation was obtained by oxidation at 150℃ for 72 h (Chang et al., 2017). Both *DI* water and medium saline water was used in this work. The medium saline water was prepared in the lab by adding a certain amount of inorganic salts into *DI* water to simulate the process water in coal preparation plants based on an investigation conducted by the Commonwealth Scientific and Industrial Research Organisation (*CSIRO*) (Ofori et al., 2010). The chemical composition of saline water is presented in Table 1. Diesel was obtained from a Shell gas station and used as a nonpolar collector. The non-ionic surfactant Triton X-100 ($C_{14}H_{22}O(C_2H_4O)_n$, n=9-10) was supplied by Sigma Aldrich and used as a polar collector in coal flotation. Methyl Isobutyl Carbinol (*MIBC*) was

supplied by Sigma Aldrich and used as a frother in coal flotation. Tween-80 ($C_{24}H_{44}O_6(C_2H_4O)_n$) was provided by Aladdin and used as an emulsifier to prepare diesel emulsion. Triton X-100, MIBC, and Tween-80 were of analytical grade and diesel was of industrial grade. All the chemicals were used in their as-received state without further purification.

Table 1. The composition of saline water used in this work (mg/dm³)

Ca^{2+}	Mg^{2+}	Na^+	K^+	HCO_3^-	SO_4^{2-}	Cl^-
93.0	126.8	1177.4	20.9	835.1	1176.8	1020.3

2.2. Methods

2.2.1. Flotation experiments

Flotation experiments were conducted in a 1.5 dm³ JK batch flotation cell with both *DI* water and saline water. For each experiment, 100 g coal was added into 1.5 dm³ water and conditioned at 900 rpm. During the flotation process, the air flow rate was kept as 3.0 dm³/min, and the pH of the coal slurry was fixed at 8.0. Triton X-100 was added first, followed by diesel and *MIBC* (10 mg/dm³). The conditioning time of Triton X-100, diesel, and MIBC was 3 min, 3 min, and 2 min, respectively. Four froth products were collected after a cumulative flotation time of 1 min, 2.5 min, 5 min, and 10 min, respectively. The froth products and flotation tailings were filtered, dried at 80℃, and weighed to calculate the flotation yields. Each flotation experiment was repeated 3 times and an average value was reported.

2.2.2. Diesel adsorption measurements

At first, oxidized coal samples were conditioned with diesel overnight to reach adsorption equilibrium, followed by the centrifugation of coal suspension to achieve solid-liquid separation. After that, the adsorbed diesel was ultrasonically extracted from coal particles with hexane being used as an extractant. Then, the concentration of hydrocarbons in hexane was analyzed by gas chromatography-flame ion detector (*GC-FID*). The measurement details have been reported elsewhere (Chang et al., 2019). Both *DI* water and saline water was used in the measurement of diesel adsorption density. The gas chromatography-mass spectrometry (*GC-MS*) analysis demonstrates that diesel is a mixture of hydrocarbons with the number of carbon atoms ranging from 11 (undecane, C11) to 25 (pentacosane, C25), and the middle hydrocarbons (e.g. hexadecane, C16) have a bigger ratio than the short hydrocarbons and long hydrocarbons. Additionally, hydrocarbons with different lengths in diesel have similar adsorption behaviour on the coal surface since they are the homologous series. Therefore, C16 was chosen to represent hydrocarbons in diesel and investigate diesel adsorption on coal surfaces. The adsorption density of C16 on oxidized coal was calculated using Eq. 1:

$$q_e = \frac{C_e \times V}{m} \tag{1}$$

where q_e (mg/g) is the adsorption density of C16, C_e (mg/dm³) is the concentration of C16 in the extracted solution, V (dm³) is the volume of extracted solution, and m (g) is the mass of coal sample. Each measurement was repeated three times before an average value was reported.

2.2.3. Zeta potential measurements

The zeta potential of diesel droplets and coal particles was measured using the Malvern Zeta Sizer (3000 E, UK) in both *DI* water and saline water by measuring the electrophoretic mobility of diesel droplets and coal particles between electrodes. When the charged solids are suspended in the aqueous phase, they are driven to move towards the oppositely charged electrodes by electrostatic forces. The zeta potential of oil droplets is calculated through the Henry equation (DC Henry, 1931) as follows:

$$\delta = 1.5 \times U_E \times \frac{\eta}{\varepsilon \times f(K_a)} \tag{2}$$

where δ is the zeta potential of diesel droplets, U_E is the electrophoretic mobility, η is the viscosity of the aqueous phase, ε is the dielectric constant, and $f(K_a)$ is Henry function.

To measure the zeta potential of diesel droplets, diesel emulsion was prepared by mixing 0.1 cm³ diesel with 1 mg Tween-80 in 100 cm³ *DI* water or medium saline water through vigorous stirring. The measurement was carried out once diesel emulsion preparation was completed. To measure the zeta potential of coal particles, 0.1 g coal particles (< 2 μm) were added in 100 cm³ *DI* water or medium saline water and conditioned at 900 rpm for 10 min. After that, the fine coal suspension was settled for 10 min before the supernatant was taken for the zeta potential measurements. Each measurement was repeated three times before an average value was presented. Error bars were calculated based on the statistical analysis of measurement results at a confidence level of 95%.

2.2.4. Diesel droplet size measurements

The effect of Triton X-100 on diesel emulsification was investigated by measuring the size distribution of diesel droplets with and without Triton X-100. The concentration of Triton X-100 used in this study ranged from 0 to 1.10^{-4} mol/dm³ to be consistent with the concentration of Triton X-100 in coal flotation. To prepare diesel emulsion, diesel, and Triton X-100 was mixed in 200 cm³ *DI* water or saline water through vigorous stirring. Once the preparation of diesel emulsion was completed, the size distribution of diesel droplets was measured using Malvern Mastersizer 2000. Each measurement was repeated three times and an average value was reported.

2.2.5. Triton X-100 adsorption measurements

The adsorption amount of Triton X-100 on the oxidized coal sample in both *DI* water and saline water was measured using Aqua Mate 800 UV-Vis Spectrophotometer. For each measurement, 1 g oxidized coal particle and Triton X-100 were added to the glass flask with 50 cm³ *DI* water or saline water. The concentration of Triton X-100 ranged from 6.5 mg/dm³ to 195 mg/dm³. The adsorption process was conducted at 200 rpm for 2 h in an incubator. After that, the mixture was transferred into the centrifuge tube and centrifuged at 6000 rpm for 10 min. Once the solid-liquid separation was completed, the concentration of residual Triton X-100 was measured using the UV-Vis spectrophotometer by measuring the absorbance value at 230 nm where the characteristic peak of the benzene ring can be found (Marsh et al., 2005). The adsorption amount of Triton X-100 was calculated as follows:

$$q_e = \frac{(C_i - C_e) \times V}{m} \tag{3}$$

where q_e (mg/g) is the adsorption amount of Triton X-100, C_i (mg/dm³) is the initial concentration of Triton X-100, C_e (mg/dm³) is the residual concentration of Triton X-100, V (dm³) is the volume of water, and m (g) is the mass of coal particle. Each measurement was conducted with two replicates and an average value was reported.

3.   Results and discussion

3.1. Flotation of oxidized coal with diesel as collector only

In mineral flotation, the overall flotation yield consists of true flotation yield and entrainment yield. It has been reported that the entrainment flotation yield in saline water was higher than that in *DI* water (Liu and Peng, 2014; Wang and Peng, 2014). In the flotation of oxidized coal, surface oxidation only affects true flotation by governing the hydrophobicity of coal particles while has not influence the entrainment process. In this work, to rule out the effect of water salinity on entrainment, the true flotation yield was determined by subtracting entrainment yield from overall flotation yield. In the previous work (Chang et al., 2018), the entrainment yield of coal oxidized 13.20 at.% in *DI* water and saline water was found to be 8.04% and 18.80%, respectively.

Fig. 1 shows the true flotation yield of oxidized coal as a function of diesel concentration in *DI* and saline waters. As shown in Fig. 1, the true flotation yield of oxidized coal in saline water was higher than that in *DI* water in the absence of the collector. This is in line with the previous finding that coal flotation in saline water has a higher tolerance to coal surface oxidation than that in *DI* water (Chang et al., 2018). With the addition of diesel, the increase of true flotation yield in saline water was higher than that in *DI* water. As can be seen, when diesel concentration increased from 0 to 80 mg/dm³, the true flotation yield in *DI* water increased slightly from 4.56% to 26.13% while the true flotation yield in saline

water increased from 11.98% to 41.99%. Therefore, the increase of true flotation yield of oxidized coal in saline water was higher than that in *DI* water, suggesting that saline water could promote the flotation of oxidized coal when diesel was used as the only collector.

To investigate the flotation behaviour of oxidized coal in-depth, the effect of saline water on the diesel adsorption was studied. Figure 2 shows the adsorption isotherms of C16 on the oxidized coal sample in the fresh and saline waters. In general, the adsorption amount of C16 on oxidized coal in saline water was higher than that in *DI* water even at very low concentration (as shown in the insert of Fig. 2), indicating that diesel adsorption on oxidized coal was enhanced by saline water. Since diesel adsorption is critical to the hydrophobicity of oxidized coal, it is inferred that the higher adsorption efficiency of diesel on oxidized coal in saline water was responsible for the improved flotation performance.
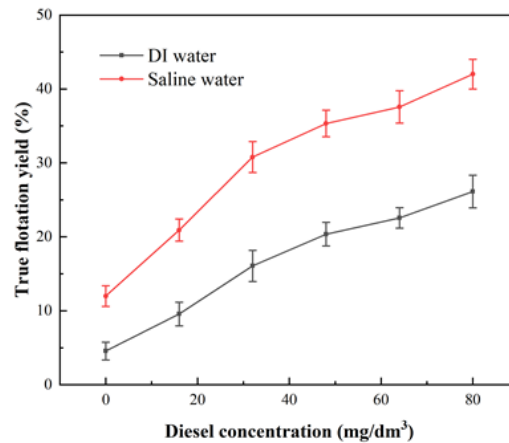

Fig. 1. The true flotation yield of oxidized coal as a function of diesel concentration in *DI* water and saline water (The data are shown as means ± sd (n=3) with 95% confidence intervals)
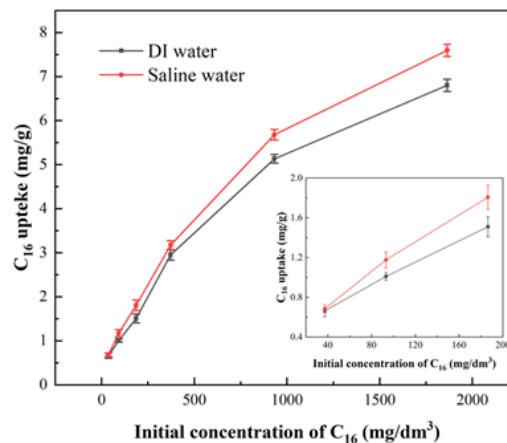

Fig. 2. Adsorption isotherms of C16 on oxidized coal in fresh and saline waters (The data are shown as means ± sd (n=3) with 95% confidence intervals)

Normally, the adsorption of diesel primarily depends on the wettability of coal surfaces. However, the previous work revealed that saline water did not increase the hydrophobicity of oxidized coal (Chang et al., 2018), thus there must be some other factors promoting diesel adsorption in saline water. In literature, it was reported that the adhesion of oil droplets on solids is governed by the interplay between oil droplets and solids through various forces, including long-range van der Walls force, electrostatic force, and hydrophobic interaction (Laskowski, 2001). Since fuel oils are usually negatively charged in water due to the preferential adsorption of OH-, the electrostatic interaction between oil droplets and solids must be considered in oil adhesion (Busscher et al., 1995; Hsu and Nacu, 2003). In general, the electrostatic attraction enhances oil adhesion while the electrostatic repulsion inhibits oil adhesion. Figures 3a and 3b show the zeta potential of diesel droplets and oxidized coal as a function

of pH in *DI* water and saline water. As can be seen in Figure 3 that the absolute value of the zeta potential of diesel droplets and oxidized coal in saline water was much smaller than that in *DI* water and much closer to nil, suggesting that saline water screened the surface charge of diesel droplets and oxidized coal by compressing their electric double layers. Therefore, it was inferred that in coal flotation where pH was 8, the electrostatic repulsion between diesel droplets and oxidized coal was reduced in saline water, thus promoting the adsorption of diesel on oxidized coals.
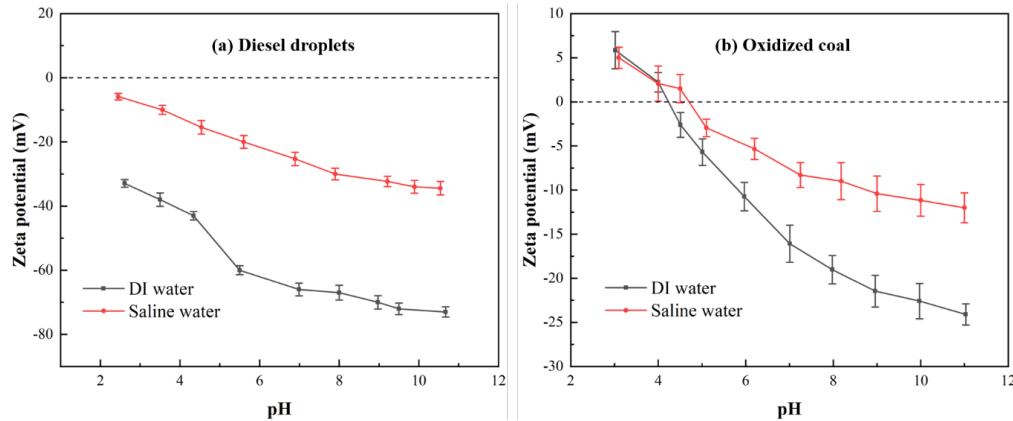
Fig. 3. Zeta potential of diesel droplets (a) and oxidized coal (b) as a function of pH in *DI* water and saline water (The data are shown as means ± sd (n=3) with 95% confidence intervals)

In summary, it can be concluded that due to the reduction of electrostatic repulsion between coal particles and diesel droplets, the adsorption of diesel on oxidized coals was promoted in saline water, which resulted in the higher true flotation yield.

3.2. Flotation of oxidized coal with diesel and surfactant as a composite collector

As shown in Fig. 1, the true flotation yield of oxidized coal was still low in saline water even at high diesel concentration, which means that a new collector system is required to improve the flotation of oxidized coal. Figure 4 shows the true flotation yield of oxidized coal in *DI* water and saline water as a function of Triton X-100 concentration. The concentration of diesel was fixed at 16 mg/dm^3 which has been proved to be sufficient to cover the un-oxidized coal surface area (Chang et al., 2017), and the concentration of Triton X-100 varied from 0 to 34.6 mg/dm^3. As can be seen from Fig. 4. that the true flotation yield of oxidized coal increased with the concentration of Triton X-100 in both *DI* water and saline water, and the true flotation yield in saline water was higher than that in *DI* water. To achieve a true flotation yield of 60%, the concentration of Triton X-100 required in *DI* water and saline water was 26.0 mg/dm^3 and 24.5 mg/dm^3, respectively.
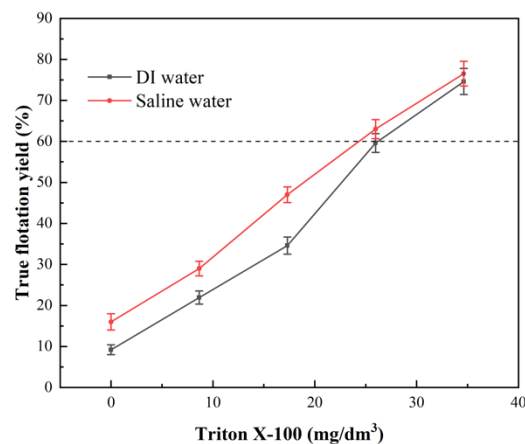

Fig. 4. True floatation yield of oxidized coal as a function of Triton X-100 concentration in *DI* water and saline water (Diesel: 16 mg/dm^3, MIBC: 6.7 mg/dm^3) (The data are shown as means ± sd (n=3) with 95% confidence intervals)

Fig. 5 shows the adsorption density of C16 on oxidized coal in *DI* water and saline water. As seen in Fig. 5, in both *D*I water and saline water, the adsorption density of C16 in presence of Triton X-100 was higher than that in absence of Triton X-100, suggesting that Triton X-100 was able to improve the adsorption of diesel on oxidized coal, regardless of water salinity. This may result from diesel emulsification by Triton X-100 which increased the contacts between diesel droplets and coal particles (Chang et al., 2019). Additionally, it is worth noting that the adsorption density of diesel in saline water was higher than that in *DI* water with or without Triton X-100, suggesting that saline water was able to promote diesel adsorption on oxidized coals, which was not influenced by the presence of Triton X-100.
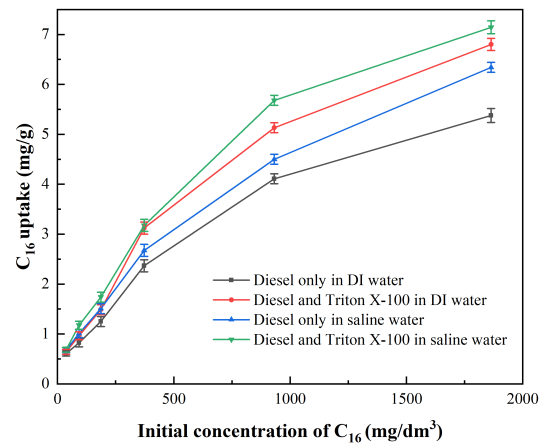

Fig. 5. Adsorption isotherms of C16 on oxidized coal in *DI* water and saline water in the absence and presence of 6.5 mg/dm³ Triton X-100 (The data are shown as means ± sd (n=3) with 95% confidence intervals)

Fig. 6 shows the average size of diesel droplets, d_{50}, as a function of Triton X-100 concentration in *DI* water and saline water. The concentration of Triton X-100 ranged from 0 to 64.6 mg/dm³, which covered the dosage of Triton X-100 used in flotation. As seen in Fig. 6, the size of diesel droplets decreased rapidly with the increasing concentration of Triton X-100 in both *DI* water and saline water. This decrease slowed down when the concentration of Triton X-100 was more than 1.23 mg/dm³, and finally levelled off when Triton X-100 concentration reached 6.46 mg/dm³. The curve in saline water was very similar to that in *DI* water, indicating that Triton X-100 is a good emulsifier of diesel which can increase the contacts between diesel and coal particles and facilitate diesel adsorption on oxidized coal. Saline water did not affect diesel emulsification by Triton X-100, demonstrating that higher diesel adsorption in saline water should also result from the reduction of electrostatic repulsion between diesel droplets and coal particles.
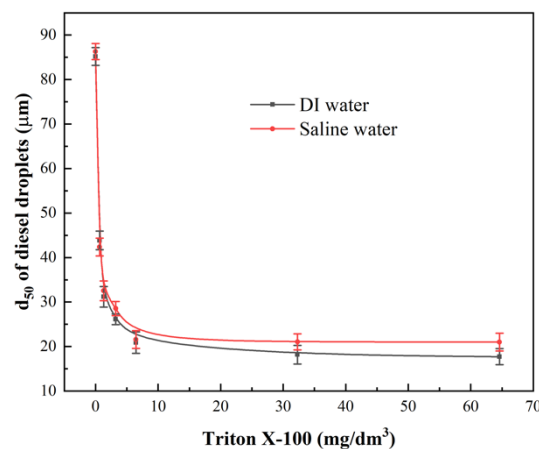

Fig. 6. d_{50} of diesel droplets as a function of Triton X-100 concentration in *DI* water and saline water (The data are shown as means ± sd (n=3) with 95% confidence intervals)

Furthermore, the results in Fig. 6 indicate that the maximum emulsification of 1600 mg/dm^3 diesel in *DI* water and saline water was achieved at 6.5 mg/dm^3 Triton X-100. Therefore, the concentration of Triton X-100 required to emulsify diesel in flotation was much smaller than 6.5 mg/dm^3. However, as shown in Fig. 4, better flotation performance of oxidized coal in *DI* water and saline water was achieved when the concentration of Triton X-100 was higher than 6.5 mg/dm^3, suggesting that the excessive Triton X-100 improved oxidized coal flotation through other ways.

Figure 7 shows the adsorption isotherms of Triton X-100 on oxidized coals in both *DI* water and saline water. As seen in Fig. 7, the adsorption isotherms of Triton X-100 in *DI* water and saline water almost coincide, indicating that water salinity did not affect the adsorption behaviour of Triton X-100. It is reported that electrolytes primarily affect surfactant adsorption by screening their electrostatic interaction with the solids or forming precipitates (Chang et al., 2018). However, Triton X-100 is non-ionic and the polyethylene headgroup can neither dissociate nor form precipitates with inorganic metal ions (Dominguez, 2009; Yu et al., 2012; Xing and Rankin, 2013; Ahmadi et al., 2014; Chang et al., 2018), which ensured that Triton X-100 could effectively emulsify diesel even in saline water. Furthermore, as reported previously, Triton X-100 can adsorb on the oxidized coal surface area through a "head-on" adsorption, which further increased the hydrophobicity of oxidized coal and improved flotation performance (Chang et al., 2017).

Above all, a schematic of the synergistic interaction between diesel and Triton X-100 in saline water is shown in Fig. 8. Diesel primarily adsorbed on the un-oxidized surface area and Triton X-100 adsorbed on the oxidized surface area with surfactant tails orienting outwards. A small amount of Triton X-100 molecules adsorbed at the oil/water interface to enhance the dispersion of diesel droplets and facilitate diesel adsorption. Compared to that in *DI* water, saline water did not influence Triton X-100 in the emulsification of diesel since Triton X-100 is non-ionic. However, the adsorption density of diesel in
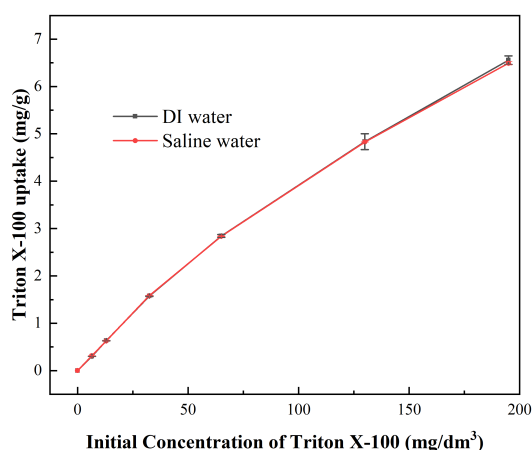

Fig. 7. Adsorption isotherms of Triton X-100 on the oxidized coal surface in *DI* water and saline water (The data are shown as means ± sd (n=3) with 95% confidence intervals)
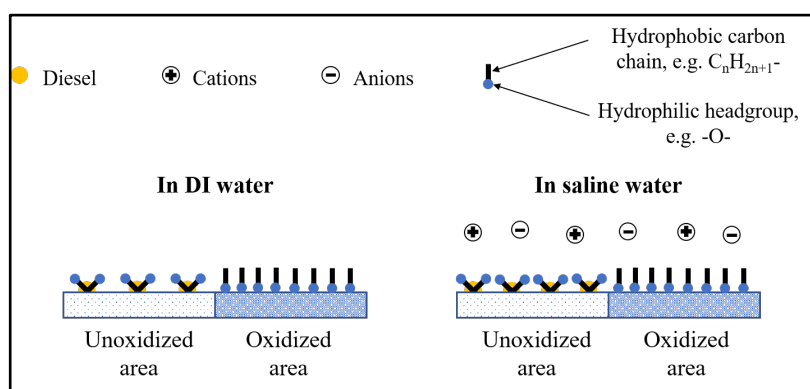

Fig. 8. Schematic of the synergistic interaction between diesel and Triton X-100 on oxidized coal surfaces in flotation in saline water

saline water was still higher than that in *DI* water, probably because of the reduction of electrostatic repulsion between diesel droplets and oxidized coal. Additionally, Triton X-100 could also adsorb on the hydrophilic oxidized surface area and further increase the hydrophobicity of oxidized coal, which is not influenced by water salinity. In summary, Triton X-100 is an effective emulsifier of diesel and promoter of oxidized coal flotation, which guaranteed the improved flotation of oxidized coal in both *DI* water and saline water.

4. Conclusions

In summary, the effect of saline water on the synergistic interaction between diesel and Triton X-100 has been studied. When diesel was used as the only collector, the true flotation yield of oxidized coal in saline water was higher than that in *DI* water, which was attributed to the enhancement of diesel adsorption due to the screening of electrostatic repulsion between diesel droplets and coal particles. However, a satisfactory true flotation yield of oxidized coal cannot be achieved in both *DI* water and saline water even at high diesel concentrations. When Triton X-100 was added with diesel as a composite collector, both the flotation yield of oxidized coal in *DI* water and saline water increased with the addition of Triton X-100, and less amount of Triton X-100 was required in saline water to achieve the same true flotation yield, suggesting that saline water could increase the effectiveness of Triton X-100 in improving oxidized coal flotation. Meanwhile, Triton X-100 could effectively emulsify diesel regardless of water salinity, thus enhancing diesel dispersion and promoting diesel adsorption on oxidized coals. Also, Triton X-100 could increase coal surface hydrophobicity through head-on adsorption on the hydrophilic oxidized coal surface area, which is not affected by saline water due to its non-ionic characteristic. Overall, Triton X-100 is an effective diesel emulsifier and promoter for oxidized coal flotation, which has good tolerance to water salinity.

Acknowledgements

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