



Use of carbon materials for produced water treatment: a review on adsorption process and performance

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Abstract

The oil and gas production is identified by consuming a large amount of water and generating massive produced water. The produced water is either reinjected into the underground layers or released into the rivers and oceans that can cause severe damage to the environment due to toxic elements such as salts, oil and grease, and polyaromatic hydrocarbons. So produced water treatment and management can reduce the significant threats to the soil and water resources and solve the lack of water in different water-consuming sectors. During the last decades, adsorption methods, such as using expanded graphite and activated carbon materials, have attracted scientists' attention because these adsorbents are cost-effective and practical. This study aimed to review expanded graphite's synthesis, adsorption process, and efficient factors in removing heavy oil, heavy metals, benzene, toluene, ethylbenzene, and xylenes, and organic acids from produced water and compare with other adsorbents, including activated carbon and residual biomass. Based on the results of extensive research works, expanded graphite's high adsorption feature suggested that graphite can be a promising adsorbent in actual produced water treatment.

Keywords Absorptive feature · Activated carbon · Expanded graphite · Heavy oil · Oil–water emulsion · Produced water

Abbreviations

AC	Activated carbon	MEG-NH ₂	Amino-functionalized magnetic expanded graphite
ACTF	Amorphous carbon thin film	MI	Microwave irradiation
Bq/L	Becquerels/L	MONPs	Manganese oxide nanoparticles
BTEX	Benzene, toluene, ethylbenzene, and xylenes	MWCNTs	Multi-walled carbon nanotubes
CTS-A-MMT	Chitosan-activated montmorillonite	NF	Nanofiltration
EG	Expanded or exfoliated graphite	O & G	Oil and grease
FGIC	Fluorinated graphite intercalation compounds	PAH	Polycyclic aromatic hydrocarbons
GAC	Granular activated carbon	pCi/L	Picocuries/L
GIC	Graphite intercalation compounds	PDMS	Polydimethylsiloxane–graphene sponge
GO	Graphite oxide	PET	Polyethylenetere phthalate
HEG	Highly exfoliated graphite	PP	Polypropylene
IFM	In situ formed magnesium hydroxide	PPQA@SiO ₂	Silica-supported polyether polysiloxane quaternary ammonium
MB	Methylene blue	PW	Produced water
MEG	Magnetic expanded graphite	RO	Reverse osmosis
		SEB	Sodium exchanged bentonite
		SEM	Scanning electron microscopy
		TDS	Total dissolved solids
		TEM	Transmission electron microscopy
		TGA	Thermal gravimetric analysis
		TOC	Total organic carbon
		TRG	Thermally reduced graphene
		WGR	Water-to-gas ratio

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WOR	Water-to-oil ratio
XRD	X-ray diffraction

Introduction

In the past decades, the oil and gas industry has accompanied by consuming a massive amount of water and generating massive wastewater during the extraction process (Jiménez et al. 2018). This wastewater, so-called produced water (PW), is reinjected into the underground layers or released into the rivers, severely affecting the environment (Igunnu and Chen 2012). Employing the untreated PW can cause numerous negative impacts on plants growth (Burkhardt et al. 2015), soil, surface water and underground water pollution (Fakhru'l-Razi et al. 2009). Discharging PW causes more threats for the oceans than other environments because PW volumes are rising in most mature offshore production fields and contain many toxic organic compounds and metals that can do permanent harm to the aquatic ecosystems (Neff et al. 2011). Given the water-to-oil ratio (WOR), the volume of PW is considerable. The average worldwide WOR is about 2 to 3 (Neff et al. 2011).

Additionally, the water-to-gas ratio (WGR) generated from a well is different in oil and gas fields. For example, the average WOR and WGR for oil and gas extraction from Federal offshore waters of the U.S. are 1.04 and 86, respectively (Clark and Veil 2009). On the other hand, the volume of generated PW is growing up to 39.5 Mm³/day. Moreover, by 2025, the WOR will be approached to 12 (v/v) for onshore crude oil resources, highlighting PW treatment's importance as an economical option for sustainable management (Arowshola et al. 2011).

PW contains various toxic organic and inorganic compounds such as salts, oil and grease (O&G), benzene, toluene, ethylbenzene, and xylenes (BTEX), polyaromatic hydrocarbons, and organic acids (Arthur et al. 2011). However, PW properties depend highly on the field's geographic location, the geological formation, the extraction method, and hydrocarbon product type. Therefore, it is vital to treat PW efficiently and change it into a less harmful by-product to meet water demands such as irrigation, livestock watering, aquifer storage, municipal and other industrial uses (Guerra et al. 2011). As a result, the primary step is to determine the significant constituents of PW. Besides, deciding PW's best treatment method plays a significant role in PW management, considering the environmental factors, economic considerations, and local regulatory frameworks (Jiménez et al. 2018).

In recent years, PW treatment has attracted scientific groups' attention worldwide. Several laboratory studies have concentrated on the treatment of PW and heavy

oil on water. However, little is known on the actual PW treatment. Igwe et al. (2013) suggested that the treatment methods involve various categories, including carbon adsorption, air stripping, membrane filtration, ultraviolet light, chemical oxidation, and biological treatment (Igwe et al. 2013). Among these, adsorption approaches are considered one of the treatments that can reach higher water qualities and the abundance of carbon-based materials, such as expanded graphite (EG), enables sorption treatments economically feasible, and there is no need for expensive equipment. Many research types in the environmental issues employed EG, a particular type of graphite, and graphene as wonder materials. The underlying reason behind this capability is that EG's active surface (around 2,630 m²/g) makes its adsorption performance higher than that of activated carbon (AC) and other materials in adsorption processes (Caniani et al. 2018). Despite EG application's importance, few review papers are presently available. A majority of these reviews discussed technologies for oil and gas PW Treatment. In contrast, little is known about adsorption processes, especially EG and AC adsorption performance and relevant comparison and discussion. Therefore, in this paper, PW's features, the synthesizing and adsorption characteristics of EG were reviewed and compared with other absorbents.

Components of PW

PW includes various chemical components affected by the depth, geochemistry of the hydrocarbon-bearing formation, the chemical synthesis of the oil and gas phases in the reservoir, chemical products added to the extraction procedure, and a lifetime of reservoirs. PW contains inorganic salts, O&G, a wide variety of organic chemicals, organic acids, primarily hydrocarbons, metals, and radioisotopes (Neff et al. 2011). The concentration of salinity in PW (changing from a few parts per thousand (‰) to 300‰) is considerably higher than that of seawater (between 32 and 36‰) (Rittenhouse et al. 1969). The presence of sodium and chloride in PW's salty contents, identified as the most abundant particles, differs between 23,000 and 141,000 (ppm) (Neff et al. 2011). The concentrations of different components, including total organic carbon (TOC), total organic acids, total saturated hydrocarbons, total BTEX, total polycyclic aromatic hydrocarbons (PAH), have been reported between 0.1–11,000, 0.001–10,000, 17–30, 0.068–578, and 0.04–3.0 (mg/L), respectively (Neff 2002). Formic acid is one of the most abundant organic acids fluctuating

from 26 to 584 (mg/L) (MacGowan and Surdam 1988). Additionally, BTEX concentration as petroleum hydrocarbons may approach 600 (mg/L) to disperse in groundwater and cause critical environmental and health issues (Su et al. 2010).

Metals are also observed in PW depending on the formations' age and geology, where the oil and gas are produced (Collins 1975). However, these metals exist in PW sample seldom. Other elements forming the PW components are radioisotopes, such as radium. It derives from the radioactive decay of uranium-238 and thorium-232 connected to certain rocks and clays in the hydrocarbon reservoir (Kraemer and Reid 1984). The concentration of radionuclides in environmental media is estimated as the rate of radioactive decay usually as picocuries/L (pCi/L) or becquerels/L (Bq/L) (Neff 2002). For example, the activity of ^{226}Ra and ^{238}U varies between 0.054–32,400 and 0.008–2.7 (pCi/L), respectively, while these amounts are 0.04 and 1.1 (pCi/L) for seawater, respectively.

Some studies presented PW characteristics (Fakhru'l-Razi et al. 2009; Jiménez et al. 2018; Neff et al. 2011). Figure 1 shows details of PW components' (Collins 1975; Neff 2002; Neff et al. 2011).

EG performance and characteristics

Literature review on the removal of heavy oil

In 1979, for the first time, a patent proposal in Japan was introduced to treat heavy oil by EG (Inagaki et al. 2001). Following that, in 1996, a Chinese group suggested a short report on the capability of using EG in the sorption of heavy oil. Their result showed that the capacity of EG sorption was 25 (g/g). Finally, in 1998, Inagaki and his group performed several studies on EG and discovered that EG could be a promising carbon-based material by adsorbed amounts in weight around 80 (g/g) of heavy oil floating on the water less than 1 min (Toyoda et al. 2000).

In a set of articles, Inagaki and his research group analyzed sorption and recovery of heavy oils using EG. Firstly, they studied the adsorption amounts using four heavy oils with different viscosities and EG with diverse bulk densities. The results showed that adsorption features depend highly on bulk density and pore volume of EG. Also, the time corresponding to maximum sorption depended on the grade of heavy oil. The authors obtained maximum sorption of 83 (g/g) within 1 min of sorption. They suggested that the higher viscous oil used in the

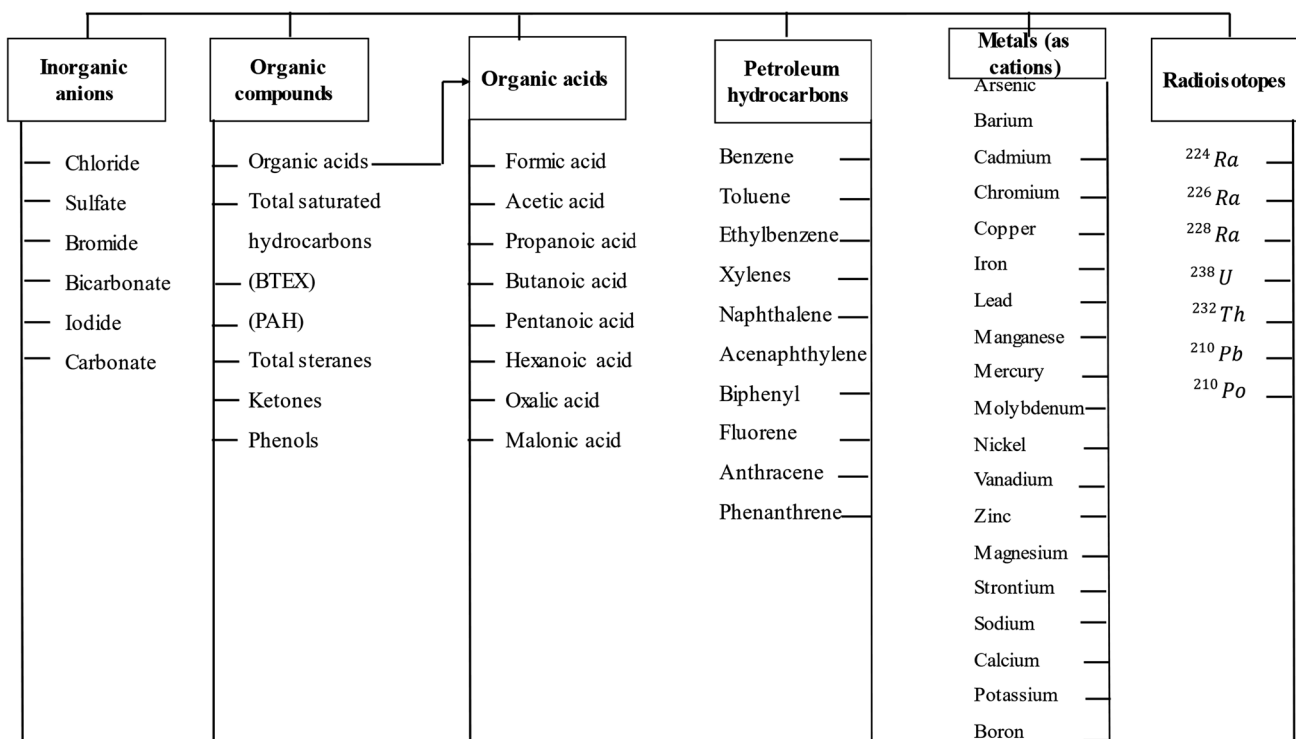


Fig. 1 Total components of PW



experiment, the more time (around 8 h) is required for the sorption process (Toyoda et al. 2000).

In another study, Inagaki et al. (2000a) evaluated the recovery of heavy oil and EG recycling with filtration following mild suction. They concluded that EG's adsorbed amounts decreased considerably with its regenerating. Moreover, the collected oils were proved to have nearly the same characteristics as the original. It is worth mentioning that the bulk density became more extensive than the initial EG's density and destroyed after squeezing. Therefore, they recommended that a much milder method is required, such as filtration in mild suction (Inagaki et al. 2000a).

In their third work, Inagaki et al. (2000b) conducted a study on the trials for functional applications by compressing EG into a plastic bag. Although this experiment aimed at reaching a notably high sorption capability by utilizing the metallic mesh and clamp, the results were roughly the same adsorbed amounts in weight as their previous work (Inagaki et al. 2000b).

Furthermore, in their fourth experiment, Toyoda et al. (2003) discussed EG's high oil adsorption. They assessed the relationship between the defined adsorbed amounts and pore size measured by a mercury porosimeter. It was the first time that such studies concentrated on evaluating the structure and pore volume of EG. They showed that huge pores, which are logically considered inter-particle pores between EG's entangled worm-like particles, were responsible for EG's high adsorption features. The volume estimated by a U-type cell illustrated an excellent correspondence to adsorbed amounts determined for A-grade heavy oil (Toyoda et al. 2003).

Finally, in 2001, Inagaki et al. (2001) considered EG usage to recover spilt heavy oil. This research aimed to compare the adsorbed amounts in weight of EG with other adsorbents. The adsorbed amounts were much larger than that of polypropylene (PP) and polyurethane mats recently used. Also, they stated that EG could sorb heavy oils preferentially (Inagaki et al. 2001). To compare the selectivity sorption of EG with other materials, they performed the same experiment on the PP mats, which were commercially accessible. Unlike EG, the PP mats adsorbed a considerable amount of water, around 4 (g/g) preferably, while this amount was 1.8 (g/g) for EG. After adding A-grade heavy oil onto the water-saturated PP mat, water was not observed to expel from the PP mat. Therefore, this feature only recognized in EG that can make it superior to other materials.

Besides, other researchers have focused on the practical usage of graphite in heavy oil and PW adsorption. For example, Bayat et al. (2008) evaluated the oil sorption

capacity of synthesized EG by current static and dynamic methods and compared it with other sorbents. They highlighted this point that graphite's thermal shock triggers the vaporization of intercalates and the decomposition of graphite intercalation compounds (GICs). This thermal shock led to an 80-fold expansion in natural graphite flakes and established the worm-like structure of EG. They showed that EG had the highest oil sorption capacity between 65 and 73 (g/g), following by PP nonwoven web with 7–9 (g/g). Furthermore, oil viscosity played a significant role in the sorption process as its higher value resulted in reducing the oil adsorption into the pores of vermicular particles and vacant spaces among the EG's entangled particles (Bayat et al. 2008).

Wang et al. (2010) employed a new magnetic characteristic to assess magnetic EG (MEG) adsorption and recovery. To control, recover and apply EG on a large scale, they set up a magnetic field around the spilt oil area; consequently, MEG adsorbed with extreme oil could immediately get together, collect and recover easily. The results showed that for MEG, the adsorption percentage raised by 18.01% and 5.65% for engine oil and crude oil, respectively. Whereas for diesel oil and gasoline, it declined by 10.95% and 4.63%, respectively, and the more probable reason was the more significant values of the glutinosity and density of engine and crude oil (Wang et al. 2010).

In another research, Iqbal and Abdala (2013) studied the use of thermally reduced graphene (TRG) in the oil spill. TRG was prepared by prompt heating of graphite, which led to an expansion of 100–300 times greater than natural graphite. In this experiment, TRG sorbed 91 g of oil A and 81 g of oil B per gram, which were more significant than the highest reported sorption capacity for carbon-based materials (Iqbal and Abdala 2013).

In practical research, scientists from Shinshu University examined the performance of EG in the oil–water emulsion. Using the micron-size iron particles enabled the easy recovery of the oil sorbed into EG from the emulsion. Considering the decrease of 100 (mg/L) of oil in the emulsion to a concentration of 0.1-few (mg/L), the performance of treatment by EG was compatible with nanofiltration (NF) or reverse osmosis (RO) membrane treatment (Takeuchi et al. 2015). Furthermore, Takeuchi et al. (2017) applied the use of EG in actual PW treatment. This study was one of the first actual PW treatment by EG. They used a laboratory-scale sorption column of EG, which could decrease the oil concentration from 278 and 66 (mg/L) to 1.2 (mg/L), and undetectable, respectively (Takeuchi et al. 2017).

Literature review on adsorption of heavy metals, BTEX and organic acids

Heavy metals removal

Figure 1 illustrates that produced water contains a wide range of heavy metals. The presence of heavy metals ions can threaten the living organism and the environment and seriously jeopardize human beings. Among heavy metals, a large amount of silver is toxic to human cells (Yurtsever and Şengül 2012). For these reasons, Ma et al. (2017) applied amino-functionalized magnetic expanded graphite (MEG-NH₂) nano-hybrids to remove Ag (I) from an aqueous solution. In this study, the influences of various factors on the performance of MEG-NH₂ nano-hybrids as an adsorbent was measured by batch experiments, such as the effects of Fe₃O₄ content in MEG-NH₂ nano-hybrids, pH, initial concentration, contact time, and dosage on adsorption properties of the MEG-NH₂ nano-hybrids. Their work reduced Ag to elemental silver in the adsorption process (Ma et al. 2017). In another experimental work, Do et al. (2019) removed lead and nickel by using expanded graphite decorated with manganese oxide nanoparticles, so-called (a newly formed composite) MONPs-EG. In this study, maximum adsorbed amounts equalled 0.278 and 0.113 mmol/g for Pb²⁺ and Ni²⁺, respectively. It suggested MONPs-EG as an adsorbent for heavy metals removal from aqueous solutions (Do et al. 2019).

In addition to EG, various studies applied expanded graphite oxide (GO) to absorb heavy metals. For example, Lee and Yang (2012) employed the flower-like TiO₂ particles on GO adsorbent to remove heavy metals ions. They showed that the growth of TiO₂ flowers on GO to form GO-TiO₂ hybrid could trigger a highly efficient adsorbent for heavy metals cations. This study suggests that adding the metal oxide (e.g., TiO₂) decoration to GO can be a promising sorbent material for eliminating heavy metals ions from aqueous solutions beyond the ordinary use of photocatalysts. The results demonstrated that the removal capacity of the GO-TiO₂ hybrid structures significantly improved as the treatment time increased from 6 to 12 h: from 44.8, 65.1, and 45 mg/g to 88.9, 72.8, and 65.6 (mg/g) for Zn²⁺, Cd²⁺, and Pb²⁺, respectively (Lee and Yang 2012). Gong et al. (2015) conducted continuous adsorption of Pb (II) and methylene blue (MB) by using GO coated sand in a fixed-bed column. The column involved a 2 cm diameter and 30 cm length glass at 25°C by adjusting four parameters, including initial concentration, flow rate, bed depth, and pH. The maximum adsorbed amounts of the GO-sand filter toward Pb(II)

was 0.63 (mg/g), whereas other adsorbents such as algae gelidium (0.083 mg/g) (Shahbazi et al. 2013), dye loaded coir fiber (0.020 mg/g) (Mondal 2009), and functionalized SBA-15 mesoporous silica with polyamidoamine groups (0.269 mg/g) (Vilar et al. 2008) adsorbed less amount of Pb than that of GO-sand. Besides, the values for MB (0.74 mg/g) was much higher than previously reported adsorbent such as cottonalk (0.024 mg/g) (Ding et al. 2014a, 2014b; Gong et al. 2015). In order to eliminate the lead, Olanipekun et al. (2014) applied GO. The adsorption rate for the various Pb concentrations (50, 100, and 150 ppm) was 98%, 91%, and 71%, respectively. They highlighted this point that the GO could outperform graphite. Therefore, it is suggested that the oxidation of AC can ease the situation to remove heavy metals in aqueous solution and portable water (Olanipekun et al. 2014). Sheet et al. (2014) performed a batch adsorption test using nanostructured GO, silica/GO composites, and silica nanoparticles to adsorb the heavy metals ions from aqueous solutions. They concluded that increasing the concentration of heavy metals in solution (200 ppm) resulted in the heavy metals' lowest elimination rate. However, in 30 ppm of nickel, cadmium, zinc, lead, and chromium, the removal rate was 89.90, 88.33, 85.60, 85.0 and 63.0%, respectively (Sheet et al. 2014).

BTEX removal

BTEX is more likely to cause severe damage to human health and aquatic and other creatures' lives (Aivalioti et al. 2012b). Owing to these life-threatening effects, eliminating BTEX from various water resources such as groundwater, wastewater, and PW has become an urgent issue. Regarding the possible recovery and regeneration of the adsorbent and adsorbate in the adsorption process, it plays a prominent role as treatment options for the elimination of BTEX from the aqueous solution (Aivalioti et al. 2012a). An extensive range of studies has been carried out on adsorption of BTEX from aqueous solutions by employing various adsorbents such as zeolite (Seifi et al. 2011; Szala et al. 2015), resins (Makhathini and Rathilal 2017, 2018), carbon nanotube (Lu et al. 2008; Su et al. 2010, 2016; Yu et al. 2016), organoclay (Jaynes and Vance 1996; Nourmoradi et al. 2012; Vianna et al. 2005) and AC (Aleghafouri et al. 2015; Daifullah and Girgis 2003).

Li et al. (2010) evaluated the application of three EG-based complex materials for removing toluene. This study's main goal was to determine proper oil from three kinds of generally used oils (plant oil, animal oil, and mineral oil) to result in the complex absorbent.



The adsorbed amounts of oils loaded on EG for 1 g of pure plant oil, animal oil, and vaseline were 1068, 966, and 817 mg/g, respectively. Li et al. (2010). Yu et al. (2016) applied magnetic multi-walled carbon nanotube (MWCNTs) nano-composite (APCNT-KOH) for adsorption of toluene, ethylbenzene, and xylene. The results showed that the APCNTs-KOH composites provided high adsorbed amounts under specific condition about 227.05, 138.04, 63.34, 249.44, and 105.59 (mg/g) for ethylbenzene, m-xylene, o-xylene, p-xylene, and toluene (Yu et al. 2016).

Organic acids removal

Kamio et al. (2004) examined the application of highly porous PEI chitosan beads (PEI-Ch) to adsorb the organic acids (acetic acid, formic acid, and pyroglutamic acid) from the fermentation process, sub-critical water hydrolysis process, and other industrial means. They suggested that organic acids transport in the particle by surface and pore diffusions (Kamio et al. 2004). In another experiment, Morad et al. (2014) studied acetic acid adsorption into AC. They employed the batch methods to estimate the contact time (4 h to 16.5% in removal rate). They concluded that the adsorption rate increases with higher initial concentration and the adsorbed amount of acetic acid drop with more AC (the optimal mass of AC was 0.5 g) (Morad et al. 2014). Pradhan et al. (2017) investigated the adsorption of lactic acid (< 10 g/L) into various adsorbents, including granular activated carbon (GAC), weak (Reillex® 425, or RLX425), and strong (Amberlite® IRA-400 or AMB400) base anion exchange resins by applying different operating conditions. The results showed that GAC, AMB400 and RLX425 adsorbed amounts were 38.2, 31.2 and 17.2 (mg/g) (Pradhan et al. 2017). Furthermore, other adsorbents such as magnetite nanoparticles (Tombácz et al. 2013) alginate/clay (Achazhiyath Edathil et al. 2020) were used.

Experimental details of EG synthesizing

The exfoliation of graphite is one of the most excellent ways to create large-scale production at a comparably cheap cost. Gaining profound knowledge of the exfoliation mechanism is significant in achieving high-quality graphene by optimizing exfoliation techniques. Some reviews are provided in exfoliation graphite (Yi and Shen 2015). EG is usually produced by the thermal shock of chemically intercalated graphite (Yakovlev et al. 2006). Besides, GICs are obtained by inserting

different chemical compounds (intercalant) between layers in natural graphite flakes (Dresselhaus and Dresselhaus 2002). Several parameters affect the process of exfoliation, including (1) the nature of graphite flakes utilized for GIC preparation, (2) the intercalant features, (3) the heating transfer performance, (4) the heating rate (Makotchenko et al. 2011). Sulfuric acid is one of the prevalent EG precursors used by adding to GIC and in the presence of an oxidizer such as HNO₃ (Inagaki et al. 2004), H₂O₂ (Tryba et al. 2003), KMnO₄, FeCl₃ (Hristea and Budrugaec 2008), O₃, Cl₂ (Avdeev et al. 1996). GIC with nitric acid is another commonly used precursor (Yakovlev et al. 2006). Furthermore, other studies suggested bromine (Chung 1987) and formic acid (Kang et al. 1997) as other precursors for EG preparation.

Various equipment and experiments are employed to assess the morphology of EG and illustrated the performance of the exfoliation process, such as powder X-ray diffraction (XRD), Raman spectroscopy, mercury porosimetry, elemental analysis, transmission electron microscopy (TEM), scanning electron microscopy (SEM), nitrogen adsorption–desorption, thermal gravimetric analysis (TGA), and dilatometer (N-type cell and U-type cell). Different studies proposed various procedures for EG synthesizing. For example, Bayat et al. (2008) prepared EG by immersing the flakes of graphite in a mixture of sulfuric and nitric acids with a 4:1 ratio for 24 h at room temperature. Prepared GIC was then cleaned with distilled water to reach a pH between 3 and 4 and stored in an oven at 100 °C for 1 h. Finally, residue GIC was converted to EG by thermal shock at 1000 °C for 5 s (Bayat et al. 2008).

Furthermore, adding iron particles facilitates the preparation of MEG as Wang et al. (2010) dissolved iron nitrate, cobalt nitrate and citric acid completely in a beaker according to the molar ratio of 1:2:4. Then, aqueous ammonia was added to obtain pH 7.0–8.0. Next, the beaker was put on the water bath at 90 °C, and the mixture solution was stirred vigorously. The weighted EG was added, and aqueous ammonia was applied to modify and keep the pH value of 7.0 to 8.0. The stirring was stopped until many colloids appeared. The sample was dried at 100 °C for 12–15 h to eliminate whole water in the next step. Finally, MEG was taken through prompt calcination in a muffle furnace at 900 °C for 90 s. However, vigorous stirring can destroy the structure of EG (Wang et al. 2010). In another study, Takeuchi et al. (2015) argued that a superparamagnetic nature of EG adjusted with iron particles facilitates the use of magnetic fields for recovering EG particles filled with oil from the treated emulsion. They prepared MEG through



2 steps. Firstly, EG was immersed in the iron citrate, dissolved in water ($\sim 90^\circ\text{C}$). To set iron particles in the EG structure, EG was dried at 600°C under a hydrogen atmosphere. Secondly, graphite-iron chloride intercalation compounds were provided by adding EG to iron chloride in a glass tube under vacuum and heat-treated at 300°C for 24 h. The samples were then dried at 600°C under a hydrogen atmosphere (Takeuchi et al. 2015).

In a recent experiment, Ghasemi et al. (2019) employed 8.5 g of CrO_3 and 7 mL of HCl for exfoliation of 1 g of graphite. Using a magnetic stirrer, 100 mL of the aqueous solution was stirred at room temperature for 2 h. Then, the solution was washed with distilled water. Finally, in the presence of H_2O_2 as an oxidizer, spread chromium into the molecular structure of graphite led to graphite expansion (Ghasemi et al. 2019). Pham et al. (2019) used a new method, so-called microwave irradiation (MI) to exfoliate EG. In this method, they mixed graphite flake, KMnO_4 , HClO_4 and $(\text{CH}_3\text{COO})_2\text{O}$ in weight ratios of 1:0.5:1:0.4 (g/g) for 10 (s) to generate GICs. Then, GICs has been placed in a 360 (W) microwave oven for less than 1 min. The final EG was named EG-MI. Also, they arranged conventional EG to compare with their new approach. For the conventional method, 1 g of graphite flake was blended with a 20 mL mixture of H_2SO_4 (intercalating agent) and H_2O_2 (oxidizing agent). The mixture was steadily washed with water on the Buchner funnel till reaching pH 5–6. The mixture was filtered and heated at 80°C . Finally, it was dried at the furnace at 1100°C for 1 min. Their result showed that the MI approach provides a greater surface area and micropore volume than the conventional method, leading

to higher adsorbed amounts for heavy oil (Pham et al. 2019).

To compare the current EG from graphite nitrate with another exfoliation method, Makotchenko et al. (2011) incorporated highly EGs (HEGs) by various intercalated mixtures based on fluorinated graphite. They applied fluorinated graphite intercalation compounds (FGICs) (40–50 mg) in a quartz tube ($d = 30\text{--}40\text{ mm}$, $h = 200\text{--}300\text{ mm}$) following by heating in a furnace about 800°C for 30 s. After the thermal shock, the HEG resulted in a much smaller number of graphene sheets than the usual EG prepared from graphite bisulfate or nitrate. Producing HEG led to increases in interlayer distances, specific surface areas ($370\text{ m}^2/\text{g}$) as well as lower bulk densities of 0.4–0.7 (g/L), which triggered higher adsorbed amounts (Makotchenko et al. 2011).

Another way to prepare adsorbents material based on graphite is by using TRG, applied by Iqbal and Abdala (2013). In this experiment, they put graphite (5 g) in a mixture of H_2SO_4 (90 mL) and HNO_3 (45 mL) inside an ice-cooled flask. Then, potassium chlorate (55 g) was added gradually to the cold reaction compound. After 96 h, the reaction finished by pouring the reaction compound into water (4 L). To clean the produced GO an HCl solution (5%) was applied until all sulfite ions disappeared. After that, the compound was then washed with water until the disappearance of all chloride ions. Following that, GO was dried in a vacuum overnight. Finally, GO was expanded by prompt heating around $1,000^\circ\text{C}$ in a tube furnace (Iqbal and Abdala 2013).

Table 1 Process of adsorption of EG

References	Method	Oil pollutants (g and L)	Water contents (mL)	EG/MEG weight	Time for complete sorption
Inagaki et al. (2000a), Toyoda et al. (2000),	Static	98 g—grade A oil	450	1 g—EG	1 min
Toyoda et al. (2003)	Static	17.2 g—heavy oil	450	Varying	Varying
Bayat et al. (2008)	Static and dynamic	Various oils	600	1 g—EG	Gas—oil 1 min Light oil 5 min Heavy oil 90 min 5 min for dynamic
Wang et al. (2010)	Dynamic	Various oils	—	—	1, 2, 10, 30, 60 min
Iqbal and Abdala (2013)	Static	10 mL—grade A oil	50	100 mg-TRG	1 min
Takeuchi et al. (2015)	Dynamic	1L—engine oil—water	varying	60 min	
Ghasemi et al. (2019)	Static	0.5 g—oil	100	0.02 g—EG	few seconds
Pham et al. (2019)	Static	Various heavy oil	—	—	6 min
Ding et al. (2014a, 2014b)	Static	Crude oil	—	1 g—MEG	—
Lutfullin et al. (2014)	Static	Petroleum products	—	Varying powder and compressed pellets of EG	15 min



Adsorption process

The adsorption process of oil and PW into EG or MEG is alike in several studies. Many researchers applied a static mode to perform the adsorption process. In this method, a specified amount of oil pollutants/PW is added to a beaker containing distilled water. Then, EG is weighed and added to the beaker. The time for performing sorption depends on the experiment (Bayat et al. 2008). In the dynamic method, the whole system is shaken for some minutes. For both methods, the oil-saturated EG or MEG is brought out. Then it is drained for some minutes and placed in a vacuum filtration setup, and weighed (Iqbal and Abdala 2013). The adsorbed amount is defined by the risen weight of EG or MEG after adsorption with Eq. 1:

$$\text{Adsorbed amounts in weight} = \frac{m_2 - m_1}{m_1} \quad (1)$$

where m_1 is EG or MEG's weight before the oil adsorption (g), m_2 is EG or MEG's weight after oil adsorption (g). Furthermore, another definition for adsorbed amounts and removal efficiency is presented by Eqs. (2) and (3).

$$Q = \frac{(C_i - C_f)V}{m} \quad (2)$$

$$P = \frac{(C_i - C_f) * 100\%}{C_i} \quad (3)$$

where Q is adsorbed amounts in given situations (mg/g), P is removal efficiency, C_i and C_f are the initial and final concentration of adsorbate in solution (mg/L), respectively, V is the volume adsorbate added in aqueous solution (mL), and m is the dosage of the adsorbent (mg). Table 1 exhibited the process of adsorption that is reported in recent researches.

Besides the typical static and dynamic process, there are other methods to conduct the adsorption. For example, Inagaki et al. (2000b) applied a different adsorption process using plastic bags filled by EG with various bulk densities. Also, they employed some polymers in the production of the bags (polyethylenetere phthalate (PET), PP, acrylonitrile and rayon). Then, the bags were immersed directly in the heavy oil. Two methods were employed to draw out the bag after the sorption of heavy oil. Firstly, they brought the bags out using stainless steel mesh, followed by hanging up the bags by using a clamp (Inagaki et al. 2000b).

Furthermore, Takeuchi et al. (2017) employed an EG column for actual PW adsorption. In a cylinder made of

PP, with an internal diameter of 35.6 mm, 3.4 g of EG were packed. The whole EG column was a permeable bed with an apparent density of 1.1 (g/cm³) with a height of the EG layer around 50 mm. Then water–oil emulsion was passed through the EG column, and the resulted effluent was collected in a glass bottle until the collected volume reached 100 ccs (Takeuchi et al. 2017).

Effective factors in adsorption performance

Bulk density and pore volume

One of the most notable parameters affecting the adsorption features of EG is the bulk density. Many studies reported a remarkable decrease in adsorbed amounts due to a higher bulk density. The amount of this reduction relates to the amount of bulk density. The principal reason for this can be the decrease in pore volume in higher bulk densities. Another worth mentioning point is that the structure of the pore volume of EG affects the adsorbed amounts. For example, Iqbal and Abdala (2013) highlighted this point that it is understandable to assume that big pores (> 50 nm) are efficient for oil sorption along with the surface area of graphene (Iqbal and Abdala 2013). In other words, the big particles in heavy oils are sorbed into macropores in EG with capillary condensation (Toyoda et al. 2000). Inagaki et al. (2001) presented that the adsorbed amount is linearly related to EG's total pore volume. So they concluded that a sufficient pore size for sorption of heavy oil is between 1 and 600 m (Inagaki et al. 2001). This EG's particular morphology is regarded as a characteristically balloon-like texture (Inagaki et al. 2000a), which is reported in almost all research in the adsorption field. Furthermore, this balloon-like or worm-like feature of EG is reported that still witnessed even after sorption of heavy oil and its desorption either by filtration following suction or washing with xylene (Inagaki et al. 2000b). This worm-like EG structure is due to the thermal shock that triggers intercalating vaporization (Bayat et al. 2008). Wang et al. (2010) showed that the myrmekitic textures and the multilevel pore structures of MEG were well-expanded compared with that of EG. However, the destruction of the characteristic worm-like morphology during the preparation by rigorous stirring resulted in a lower absorption capacity (Wang et al. 2010). Therefore, selecting an effective adsorption method plays a vital role in adsorption studies concerning the worm-like morphology.



Heavy oil characteristic

The viscosity of heavy oil or oil contents in PW is another effective parameter influencing the adsorption rate. Several studies reported that more viscous oil is needed a longer time to be adsorbed (Wang et al. 2010), for example, 8 h (Toyoda et al. 2000). Besides the adsorption time, the amount of oil absorbed into the vacant spaces between EG's entangled particles is affected by oil viscosity. Bayat et al. (2008) stated that the sorption capacity of EG reduces as oil viscosity increases. (Bayat et al. 2008). Iqbal and Abdala (2013) explained that the higher viscosity of heavy oil related to the higher concentration of long-chain hydrocarbons in high viscous oil (Iqbal and Abdala 2013).

Regeneration of EG and oil recovery

Recycling both heavy oil and EG is necessary and economically cost-effective. However, the adsorbed amounts in weight and volume of recycled oils decrease significantly with increasing cycling times. Inagaki et al. (2000a) stated that approximately 30 to 40% of heavy oil stuck on the surface, and the graphite flakes' pores can significantly decrease the adsorbed amounts of oil

and PW after each cycling (Inagaki et al. 2000a). Iqbal and Abdala (2013) concluded that the adsorbed amounts decreased after graphite regeneration due to an increase in the unrecovered/retained oil with cycle number and the possible compaction of TRG and decreased pore volume during the filtration/compression process (Iqbal and Abdala 2013).

The temperature of oil contents

The temperature of oil affects the adsorbed amounts. The higher-temperature triggers a less viscous oil that can result in higher adsorption. However, heavy oil recycling at high temperature is not practical because of the evaporation of light components. (Toyoda et al. 2000). Additionally, increasing the temperature of actual PW is not easy in real adsorption situations. Bach et al. (2016) conducted their experiment at a varying temperature from 10 °C to 30 °C. The results confirmed the raising of removal of diesel oil with increasing temperature. Moreover, the underlying reason behind this can be related to easier oil diffusion into the pore of EG/CoFe₂O₄ at high temperature (Bach et al. 2016).

Table 2 Adsorbed amounts of oil and PW into EG/MEG reported in recent experiments

Reference	Material	Type of oil	Bulk density of adsorbent (g/cm ³) or (g/L)	Specific surface area (m ² /g)	Total pore volume (cm ³ /g)	Adsorbed amounts in specific conditions (g/g)
Inagaki et al. (2000a), Toyoda et al. (2000)	EG	Grade A oil	0.006 g/cm ³	62	21.8	83
Inagaki et al. (2000b)	EG	Grade A oil	0.006–0.017 g/cm ³	–	–	70–80
Toyoda et al. (2003)	EG	Grade A oil	0.0087 g/cm ³	–	74	85
Bayat et al. (2008)	EG	Gas oil	0.0045 g/cm ³	–	–	73
Wang et al. (2010)	EG	Engine oil	–	225.53	–	41.61
Wang et al. (2010)	MEG	Engine oil	–	216.28	–	48.93
Iqbal and Abdala (2013)	TRG	Grade A oil	3 g/L	1,260	3.7	91
Makotchenko et al. (2011)	HEG	Benzene, sulfuric acid 1,1-dimethylhydrazine	0.4–0.7 g/L	370	–	205 (benzene), 395 (sulfuric acid) and 188 (1,1-dimethylhydrazine)
Ding et al. (2014a, 2014b)	MEG	Crude oil	–	–	–	35.72
Lutfullin et al. (2014)	MEG	Motor oil	0.03 g/cm ³	–	645	110
Takeuchi et al. (2015)	MEG	Oil–water	0.007 g/cm ³	49.7	–	Initial concentration: 100 mg/L, Final concentration: 0.1 mg/L*
Takeuchi et al. (2017)	MEG	Actual PW	0.007 g/cm ³	49.7	–	concentration: 278 mg/L, Final concentration: 1.2 mg/L**

*It should be noted that 100 mg/L of oil in emulsion decreased to a concentration of 0.1–few mg/L in this experiment. **Also lab-scale sorption columns of EG decreased the oil concentration in actual produced water from 278 to 1.2 mg/L

Carbon to oxygen (C/O) ratio

Iqbal and Abdala (2013) noted that TRG adsorption performance is highly influenced by bulk density and the C/O ratio. TRG C/O ratio, as representative of TRG hydrophobicity, greatly influences adsorbed amounts. The more TRG C/O ratio, the more TRG hydrophobicity increases the adsorption features (Iqbal and Abdala 2013). Toyoda et al. (2003) highlighted this point that the hydrophobic (oleophilic) feature of the surface of EG seems to be an underlying reason in heavy oil sorption (Toyoda et al. 2003).

Contact time

The required time to achieve the maximum adsorbed amounts under given conditions depends on the viscosity of heavy oil or contaminant (Inagaki et al. 2000b). Bayat et al. (2008) observed that gas oil was adsorbed in 1 min, whereas light crude oil and heavy crude oil needed 5 and 90 min because the heavy crude oil is more viscous (Bayat et al. 2008). Other studies reported various contact time to complete the process of adsorption. For example, after 2 min (Wang et al. 2010), 6 min (Bach et al. 2016) and 60 min (Takeuchi et al. 2015), the adsorbed amounts remained stable. However, the stabilization time varies depending on the amount of EG added.

Initial concentration of adsorbate

The effect of the initial dosage (concentration) of adsorbate on the adsorbed amounts depends on the experiment's situation. For example, Bach et al. (2016) evaluated the influence of adding various dosage of oil content (15, 20, 25, 30, and 35 g) on adsorption performance. They observed that by adding the floating oil, the amounts of adsorbed oil declined because the adsorbent outperforms in less oil dosage on the surface by providing more porosity (Bach et al. 2016). While Tuan Nguyen et al. (2019) reported that adsorption features of MnFe_2O_4 in diesel oil did not alter significantly as well as crude oil removal experienced a small fluctuation in the situation of adding a different dosage of oil (15 to 45 g) (Tuan Nguyen et al. 2019).

Salinity

Bach et al. (2016) reported that salinity (ranging from 0.5% to 3.5%) could make the process of adsorption easier because of the reduction in the solubility of diesel in

the more salinity water (Bach et al. 2016). Furthermore, the work of Tuan Nguyen et al. (2019) confirmed the effect of salinity on adsorption. They changed the NaCl concentration from 1 to 3%. The results illustrated the efficient removal of heavy oil in the saline water environment by slight fluctuation in adsorbed amounts (Tuan Nguyen et al. 2019). On the other hand, other factors can change adsorbents' performance, such as pH and adsorbent dosage. However, the effects of such parameters on oil sorption behavior need to be clearly evaluated (Takeuchi et al. 2017). Table 2 illustrates the adsorbed amounts measured under arbitrary conditions in various studies.

Other adsorbents

Besides graphite-based material, other adsorbents were widely used in oil–water and PW treatment such as AC, zeolites (Hansen 1994), resins and copolymers (Carvalho et al. 2002), bentonite (Doyle and Brown 2000), organoclay, eggshells (Muhammad et al. 2012), nutshell (Rawlins and Sadeghi 2018), chitosan (Akhbarizadeh et al. 2018; Hosny et al. 2016). Activated carbon can be produced from carbonaceous material, including coal, wood, or nutshells (for example coconut) as well as GAC is widely studied in the oil-field for removing BTEX from water produced with natural gas and with aquifer gas storage (Doyle and Brown 2000; Fakhru'l-Razi et al. 2009). Many studies proved that the residual biomass [for example papyrus reed (Al-Zuhairi et al. 2019), palm shell, sawdust, orange peel, banana peel, passion fruit peel, and cocoa beans (Gallo-Cordova et al. 2017)] could be considered as adsorbents for the eliminating the organic compounds existing in PW. However, a pre-treatment method is necessary, such as heating or washing by acid (Gallo-Cordova et al. 2017). Some of the porous polymers, such as polypropylene and polyethylene terephthalate, have been employed to absorb spilt oil that adsorbed amounts (risen in weight) is 10~20 g of heavy oil per 1 g polymer (Inagaki et al. 2001). Table 3 presents the application of various materials in PW treatment.

Discussion and conclusion

PW's reinjection into groundwater or releasing it into the oceans cause irrecoverable damage to water resources, marine organisms, and communities. Because of the increasing WOR to 12 (V/V) by 2025, PW's treatment will have much of a role to play in preventing further

Table 3 Performance of other adsorbents

References	Material	Type of pollutant	The initial concentration of oil	The final concentration of oil (after adsorption)	Oil removal percentage (%) or the amounts of adsorbed oil/pollutant (mg/g) or in weight (g/g)
<i>Activated carbon</i>					
Zhai et al. (2020)	PPQA@SiO ₂ ^a	PW	300 mg/L	23.7 mg/L	92.1%
Okiel et al. (2011)	Bentonite-AC	Oil–water	1012 mg/L	17 mg/L	98.32%
Bentil and Buah (2018)	Palm kernel shells AC	PW-chemical oxygen demand	1160.97 mg/L	108.9 mg/L	90.62%
Bentil and Buah (2018)	Coconut shells AC	PW-chemical oxygen demand	819.23 mg/L	124.04 mg/L	84.37%
Kusworo et al. (2018)	AC-bentonite	PW (total dissolved solids (TDS), turbidity, and salinity)	–	–	72%, 6%, and 90%, respectively
Carvalho et al. (2019)	AC—vegetables and animal bone	PW (TOC)	28 for vegetables and 15 mg/g		
Okiel et al. (2011)	Deposited carbon DC	oil–water	1012	465	54.1%
Fathy et al. (2018)	ACTF ^b	PW	132.77 mg condensate/g		
<i>Residual biomass</i>					
Al-Zuhairi et al. (2019)	Papyrus reed	PW	5000 ppm	275 ppm	94.5%
Okologume and Olayiwola (2019)	Banana peel	PW	40.48 mg/L	1.61 mg/L	96%
Muhammad et al. (2012)	Eggshells	PW	173 mg/g		
Gallo-Cordova et al. (2017)	Sawdust	PW	33 mg/g		
Hosny et al. (2016)	Natural polymer (chitosan) from shrimp shells	PW	3853.93 ppm	146.07 ppm	96.35%
Akhbarizadeh et al. (2018)	CTS-A-MMT ^c	PW (metals, crude oil)	–	–	93% for metals and 87% for crude oil
Ngobiri et al. (2020)	Sodium exchanged bentonite (SEB)	Xylene ethyl benzene, toluene, gasoline, kerosene, diesel, and water	Xylene 1.04, ethylbenzene 1.01, toluene 1.41, gasoline 1.27, kerosene 1.02, diesel 1.46, and water 7.02 (g/g)		
Ngobiri et al. (2020)	Organophilic clay	Xylene ethyl benzene, toluene, gasoline, kerosene, diesel, and water	Xylene 4.66, ethylbenzene 6.11, toluene 8.23, gasoline 5.06, kerosene 6.22, diesel 11.96, and water 0.01 (g/g)		
Kogiso and Aoyagi (2018)	Organic nanotubes	PW	66 mg/0.5 g		
Dong et al. (2018)	IFM ^d	PW	10.959 (g/g)		
Jang and Chung (2018)	Titanium-based adsorbent H ₂ TiO ₃	Shale gas PW (lithium)	2.58 mmol/g		
Tran et al. (2015)	PDMS ^e	PW	6.5 g/g		

^aSilica-supported polyether polysiloxane quaternary ammonium^bAmorphous carbon thin film^cChitosan-activated montmorillonite^d*In situ* formed magnesium hydroxide^ePolydimethylsiloxane–graphene sponge

consequences and supplying a vast water resource for using in various sectors such as industrial parts and irrigation demands. EG's high adsorption feature suggests that graphite can be a promising adsorbent in actual PW treatment among adsorption treatments. Conversely, the other adsorbents showed less adsorbed amounts than that of EG. For example, 91 g of A-grade oil can adsorb by 1 g of EG or 278 (mg/L) of actual PW can be reduced to 1.2 (mg/L) (99.6% removal percentage) (Table 2).

In contrast, the range of adsorbed amounts in defined conditions of other adsorbents was less than that of EG. Take IFM for an example; the adsorbed amount was around 10.5 (g/g) (Table 3). Another disadvantage of other materials is that they need a pre-treatment process making it difficult to perform large-scale applications.

On the other hand, EG provides a high specific surface area and less expensive cost. These features enable using EG as one of the most appropriate options in actual PW treatment. However, several factors affect the adsorption features under specific conditions, and they should be considered and studied carefully. For example, the bulk density of EG is the most significant factor in achieving higher adsorbed amounts of PW. Moreover, the PW adsorption process into EG is easy to be performed and does not need complicated equipment and apparatus. However, few studies about using EG column or other experiment conditions in PW are presently available.

Furthermore, the pore volume of EG affects the entire capability of the adsorbent. The worm-like morphology allows EG to adsorb the massive oil contents between vacant spaces in the entangled particles, which are not the case in other adsorbents. EG's reuse and regeneration is a unique feature that enables using it for more than one time. Conversely, the recovery of materials in other adsorbents is not practical.

This review article evaluated PW's components, EG background in adsorption studies, synthesizing the EG, adsorption features of EG and its influential factors and other adsorbents. The significant points of using different adsorbents material can be summarized as below:

- PW contains various toxic organic and inorganic chemical components that pose a danger to the environment, especially ocean ecosystems.
- In recent decades, several studies have conducted on using EG for removing oil contents of oil–water emulsion. However, few studies focused on using EG in actual PW treatment.
- Several studies have conducted on the adsorption of oil–water contents into EG. The results of these experiments indicated a considerable volume of oil removal by using EG (for example, 91 (g/g) (Iqbal and Abdala 2013)). Takeuchi et al. (2017) reduced

the 278 (mg/L) initial concentration of oil contents to less than 1.2 (mg/L) in an experiment carried on actual PW by EG column (Takeuchi et al. 2017).

- The time for achievement of adsorption of EG was reported variously. Overall, it ranges between a few seconds for light oil and 90 min for heavy oil.
- Adding a magnetic field to EG make the collection and recovery of MEG easier.
- Many factors influence EG's adsorption features, such as bulk density, pore structure, oil characteristics, recovery of EG, the temperature of oil, C/O ratio, and pH.
- It is worth mentioning that EG could adsorb heavy oils preferentially. For proving this selectivity of adsorption, Inagaki et al. (2001) performed the same experiment on the PP mats, which were commercially accessible. Unlike EG, the PP mats adsorbed a considerable amount of water, around 4 (g/g) preferably, while this amount was 1.8 (g/g) for EG. After adding A-grade heavy oil onto the water-saturated PP mat, water was not observed to expel from the PP mat. Therefore, this feature only recognized in EG can make it superior to other materials (Inagaki et al. 2001).
- In addition to heavy oil, the PW contains various components such as heavy metal, BTEX, and organic acids, severely affecting human health and putting the environment in danger. Numerous studies were carried on the removal and adsorption of these highly challenging components.
- Other adsorbents were used to adsorb PW, such as AC, residual biomass, chitosan, and bentonite. However, the amount of PW and oil adsorbed into these adsorbents was less than that of EG.

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Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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