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Treatment of oil–water emulsions by adsorption onto resin and activated carbon

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ABSTRACT

Water is essential in most of the industrial processes in order to guarantee proper production. Minimizing waste is one of the principles behind any circular economy initiative. Typically, industrial waste water contains oil and grease, BTEX (benzene, toluene, ethyl benzene and xylenes), high content in dissolved solids, suspended solids, heavy metals, biological oxygen demand (BOD) and chemical oxygen demand (COD). In order to be discharged or reused, waste water needs to be treated to meet the existing regulations. Emulsified oil in waste water constitutes a severe problem in the different treatment stages before being disposed of in a manner that does not violate environmental criteria. Several technologies are already in place to treat refinery wastewater. One commonly used technique for remediation of petroleum contaminated water is adsorption. The main objective of this study is to examine the removal of oil from oil-water emulsions by adsorption on a Dow resin and activated carbon. Experiments were performed at the Dow Water and Process Solutions (DW & PS) Global Water Technology Center (GWTC) in Tarragona, (Spain) in order to evaluate the removal of hydrocarbons from real oily water samples from a petrochemical company in Spain using DOWEX™ OPTIPORE™ L493 Polymeric Adsorbents and commercial granulated activated carbon. The results showed that the adsorbents were able to remove oil from water and that the adsorptive properties of the adsorbents are influenced by different factors such as contact time, flow rate, etc. Test data showed that OPTIPORE L493 could remove up to 99.7% of the oil-in-water contents consistently at flow rates where activated carbon efficiency had started to decline. The present paper includes the details of the results of this study.

Keywords: Hydrocarbons; Emulsified oil; Dissolved oil; Wastewater; Produced water; Activated carbon; Resin; Optipore; Adsorption

1. Introduction

Nearly all industrial processes require large amounts of water for processing, washing, diluting, cooling, sanitization or even transportation. These processes generate large amounts of wastewater that must be treated to the levels specified within the relevant regulatory framework so that the water can either be reused or discharged.

The most relevant pollutants in wastewaters from the oil processing industry are oil and grease, suspended solids, pH, BTEX, phenolic compounds, COD, sulfide and ammonia. By far, the most difficult to remove are oil and grease (O&G) contaminants. When present in contaminated wastewater discharge, O&G can cause system breakdowns, sewage spills, and environmental damage to the ecosystem through soil and aquifer contamination [1].

This paper discusses DOWEX™ OPTIPORE™ L493 Polymeric Adsorbent as a polishing technology for oil

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removal as compared to activated carbon (AC). AC is a common and simple adsorption technology used to remediate petroleum contaminated water [2]. This paper makes a side-by-side evaluation of the adsorption performance of the removal of hydrocarbons of samples from a petrochemical company by using OPTIPORE L493 and commercial granulated activated carbon. The results are used to discuss the influence of flow rate on adsorption capacity and the implications for OPTIPORE L493, a resin adsorbent especially designed for O&G adsorption, which could come to replace AC.

2. Background

2.1. Hydrocarbons in industrial wastewater

O&G is a term that refers to a variety of substances, including fuels, motor oil, lubricating oil, hydraulic oil, cooking oil, and animal-derived fats. There are two main components of O&G: petroleum-based hydrocarbons and fatty compounds of animal or vegetable origin. Oily substances will appear as free, dispersed, or dissolved oil. It is the last two, dispersed and dissolved oil, which represent the greater challenge in O&G separation. Dispersed (emulsified) oil appears as small droplets (0.5–80 µm) of oil suspended inside the aqueous phase; whereas dissolved oil comprises the polar constituents of oil, a mixture of hydrocarbons that includes BTEX, small polycyclic aromatic hydrocarbons (PAH) and phenols [3].

The test procedures used to measure fat, oil, and grease (FOG) concentrations in wastewater do not determine the presence of specific substances. The test, instead, measures entire groups of substances that can be extracted by using a particular solvent (usually hexane), regardless of origin [4]. However, FOG measurements often exclude some hydrocarbon constituents of dissolved oil. A more accurate measurement of all hydrocarbon pollutants in water is total petroleum hydrocarbons (TPH), which is a general indicator of petroleum content that accounts for all components measured by FOG methods as well as BTEX and other dissolved components [5].

2.2. Drivers for oil removal

In general, the amount of oil that must be removed from wastewater is determined by either government regulations or the need to condition water for reuse. However, facility operators typically seek to remove the oil content below the limits of classic and well-established technologies because they are attempting to remove successively both dispersed and dissolved oil.

The greatest fraction of industrial oily water pollution comes from the upstream Oil and Gas sector in the form produced water (PW) coming out of oil formations alongside oil and gas. PW is the main byproduct in Oil and Gas operations with a large oil-in-water content, producing more than 67.6 million m^3/d [6]. As the total volume of produced water grows yearly, worldwide regulations for produced water discharge into the sea are becoming ever stricter. Currently international regulations establish oil-in-water (OIW) content limit at 40 ppm [7]. However, individual nations have independently started further lowering permissible levels of oil and grease pollution for discharge: the Oslo-Paris Convention agreed to 30 ppm of OIW and set the goal of zero-discharge of OIW by 2020, the Persian Gulf has its current OIW limits at 15 ppm, and the Qatar Ministry of Energy set at zero-discharge limit for 2016 [8]. The fact that the large costs of PW treatment have become unavoidable has led many Oil and Gas companies to investigate reusing PW for pressure maintenance or even beneficial reuse. In upstream Oil and Gas, as well as in refinery wastewater, the exploration of new technologies is also driven by the possibility of recovering the valuable residual oil.

Water scarcity and concern for the impact wastewater discharge has on the environment also have contributed to the growing appreciation of water reuse. In many instances, industry is starting to consider progressive implementation of zero-liquid-discharge (ZLD) processes. This has created the need to implement de-oiling systems to protect sensitive technologies such as reverse osmosis trains and evaporators [8]. In boilers and heat exchange systems, leakages in motors and pumps regularly contaminate closed water circuits. In such cases de-oiling is undertaken for condensate polishing to prevent efficiency loss.

2.3. Adsorption technologies for oil removal

There is a wide array of technologies that can remove O&G in wastewater streams. Which technology to use depends on the concentration of oil, the target concentration, footprint, energy, etc. Adsorption is a widely accepted technology for removing trace hydrocarbons from wastewater. It is generally used only as a tertiary treatment or polishing step because most adsorbents can easily become depleted and overloaded when there are quality upsets in feed water [7]. However, adsorption can do what most other technologies –except select extraction processes like MPPE [9] – cannot: remove both fine emulsions and dissolved oil from a stream.

To achieve significant capacity as an adsorbent, materials must have a high specific area, which implies a highly porous structure comprised of micro pores (<2 nm), meso pores (2–500 nm), and macro pores (>500 nm). Additionally, the adsorption capacity of a material is defined by the presence of functional groups that can confer polar or nonpolar characteristics, with marked effect on the selectivity toward certain molecules of alike polarity [10]. The adsorption of nonpolar oil can be successfully achieved over materials like activated carbon (AC) [11], organo clays [12], zeolites [13], copolymers [14], and resins [15]. Due primarily to their low cost, ACs have historically been the most widely used material for adsorption processes in water treatment [2].

Granular activated carbon (GAC) has for many years been the de facto standard for removing soluble hydrocarbons from wastewater by adsorption. It is a highly porous organic material that is extremely good in the adsorption of organic and nonpolar molecules. Its natural hydrophobicity favors the adsorption of nonpolar molecules over water by avoiding the hydration of adsorption sites. Equilibrium adsorption on activated carbon has been amply studied separately for most types of compounds that are typically present in produced water such as PAH [16], phenolic compounds [17], vapor [18] and aqueous phase BTEX [19], and other typical short-chain hydrocarbons [20]. Where it concerns the adsorption of various petrochemicals it has been found that rates of adsorption on granular activated carbon are quite low and are greatly influenced by intra-particle diffusion, which depends on pore size distribution and molecular size. Increasing molecular weight has been shown to improve on the adsorption capacity of an activated carbon, whereas branching is known to decrease it. Furthermore the total carbon removal for a mixture is normally enhanced compared to single-solute data [21]. However, AC adsorption studies do not often use a complex multi-component oil-in-water matrix. Nonetheless there have been some studies that show that at equilibrium AC can have an adsorption capacity about 37% w/w [22], 46% w/w [23] in TPH or 55% w/w in FOG [24].

AC can be obtained from a number of sources, such as coconut shells, bituminous coal, and lignite, and can be produced in a two-step process of pyrolysis at 300–500°C followed by thermal or chemical activation [2]. Its specific properties vary depending on source material and production procedure, creating ACs with highly variable pore distribution and average specific area, running the spectrum of pores diameters of 1–1000 nm, of densities of 0.5–0.9 g/cm^3 and specific area of 200–1200 m^2/g [25].

DOWEXTM OPTIPORETM L493 Polymeric Adsorbent is a commercial adsorbent well-known for its ability to efficiently remove aromatic compounds from aqueous solution. Classically it is a resin used for the removal of furan derivatives from sugar [26], and has also been studied for the adsorption of acrylonitrile [27], fuel additives such as MTBE [28,30], alcohol biofuels [30] and phenolic compounds [31] with high removal rates. As dissolved oil is largely made up of BTEX compounds and other short-chain hydrocarbons, OPTIPORE L493 is an adsorbent with potential for produced water remediation. Indeed although there is not a large amount of literature on this application, it has been previously cited in patents as a potential polisher for oil removal systems [32] and general system for organics removal in produced water treatment [33].

OPTIPORE L493 is a highly cross-linked styrene-divinylbenzene copolymer resin with a high surface area (1100 m^2/g), an average pore diameter of 4.6 nm and apparent density of 0.62 g/cm^3 [34]. The absorption capacity for OPTIPORE L493 is estimated at 21% w/w depending on the exact composition of the mixture pounds of total petroleum hydrocarbons (TPH). OPTIPORE L493 has shown to be able to reduce TOC from 225 mg/l to 140 mg/l as well as removal of BTEX of up to 99.98% [32].

Compared to AC, a large fraction of the surface area in OPTIPORE L493 can be found in the macro pores (Fig. 1). This is important because the relative size of pores to the sorbate molecule governs the adsorption behavior [35]. Generally, micro pores make a dominant contribution to the adsorption capacity because they are barely larger than the molecules that penetrate them, and these can rarely escape the force field of the pore walls. However, the transport in micro pores can be limited by steric effects, which limits the capacity of large unwieldy molecules – such as some BTEX and PAHs – to be adsorbed. Macro pores make small contributions to the adsorption capacity but have a major impact in adsorption kinetics by making micro pores more accessible to large molecules [25].

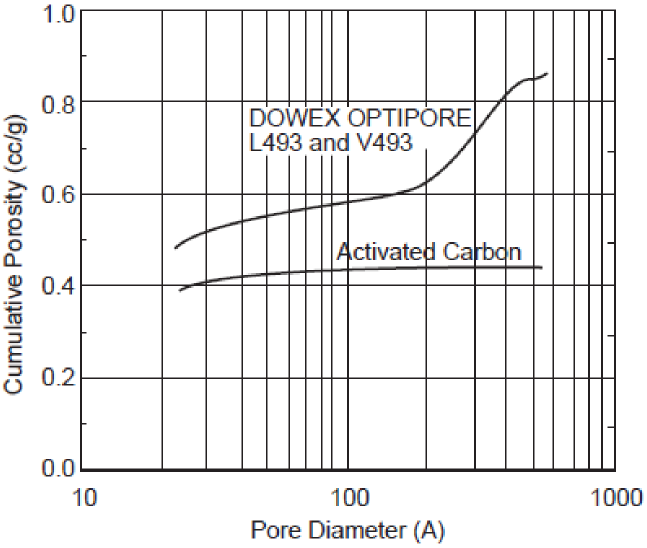

Fig. 1. Pore size distribution of activated carbon and DOWEX™ OPTIPORE™ L493.

Granular AC and DOWEX™ OPTIPORE™ L493 are comparable technologies. They are both packed into columns where the hydrocarbons present in the water will adhere onto the surface of the adsorbent and be retained within the porous structure. The major concern of an adsorption unit will be its operational adsorption capacity. While the total adsorption capacity is a measure of all available active sites for adsorption at equilibrium, operating capacity is merely a fraction – large or small – of total capacity. It depends on a number of process variables such as concentration, temperature, depth of the bed, and flowrate [36]. Depending on the sorbent, limitations exist pertaining to their operational adsorption capacity determined by their adsorption kinetics.

Because both adsorbents observe physical adsorption of oil by Van der Waals forces and induced dipole interactions, they both can be regenerated. Desorption in DOWEX™ OPTIPORE™ L493 is relatively simple and can be induced either with aqueous acids or bases, organic solvents or with steam [34] and can be performed in-situ. Often, AC is disposed of after a single absorption cycle because it is cheaper to replace than to reactivate. Typically, used AC is sent to a landfill, which could become an environmental concern; the biological degradation that occurs of the absorbed components could potentially lead to odors and groundwater contamination. In the case of granular AC in packed beds, regeneration procedures are also common [37].

There are numerous methods for reactivating AC; however, the most prevalent industrial practice is high-temperature reactivation carried out in furnaces at temperatures as high as 1000–1100°C in the presence of an inert gas [38]. Having a regeneration unit on site is expensive, and so reactivation tends to be subcontracted, which is difficult for facilities that operate in remote places, such as an offshore oil platform. Furthermore, following every regeneration cycle, there is a loss of volume and adsorption capacity per unit volume of carbon [37]. The industrial practice for AC beds is to "make up" the difference with fresh virgin carbon to total volume in a pseudo-stationary state. There are numerous other methods for AC regeneration, but each of them has its own set of problems, such as the use of volatile organic solvents for extractive regeneration or being economically inviable for long-term operation as in reactive regeneration [39].

3. Methodology

Two different adsorbents were tested in these experiments: commercial activated carbon and DOWEX™ OPTIPORE™ L493 Polymeric Adsorbent (Table 1). The experiments were conducted in two 6 cm diameter glass columns and a bed depth of 20 cm packing 500 mL of absorbent, each packed with activated carbon and OPTIPORE L493 (Fig. 2). The columns were fixed on a metal stand and a collection flask was placed underneath the column to collect the effluent. The feed flow was top-to-bottom, maintaining a constant flow rate throughout each experiment at 30, 60 and 120 BV/h, where BV/h is volume per hours of liquid to be treated per volume of resin. The set-up was fully manual, and the experiments were carried out in batch mode at ambient temperature (18–22°C) and atmospheric pressure.

For the experiments, the samples tested were real-life samples of refinery process wastewater from a petrochemical company in Spain. The samples were process water extracted from the same sample point but during different days. Historical analysis has shown that the dissolved oil may contain methanol, BTEX, styrene, cumene, naphthalene, indene and other aromatic hydrocarbons as determined by GC-MS and GM-FID methods. Additionally they carry suspended solids, heavy metals, high content of BOD and COD as well. However, the exact composition and oil-in-water concentration will show important variation depending on different operational factors. The samples tested were in the range of 250 ± 40 mg/l of TPH, which is on the higher end of the registered historical values.

Each sample was simultaneously tested in parallel with activated carbon and DOWEX™ OPTIPORE™ L493 with aliquots of 800 ml. These operation conditions in the upper range for adsorption technologies were chosen to be able to observe and quantify differences between both media in short duration experiments.

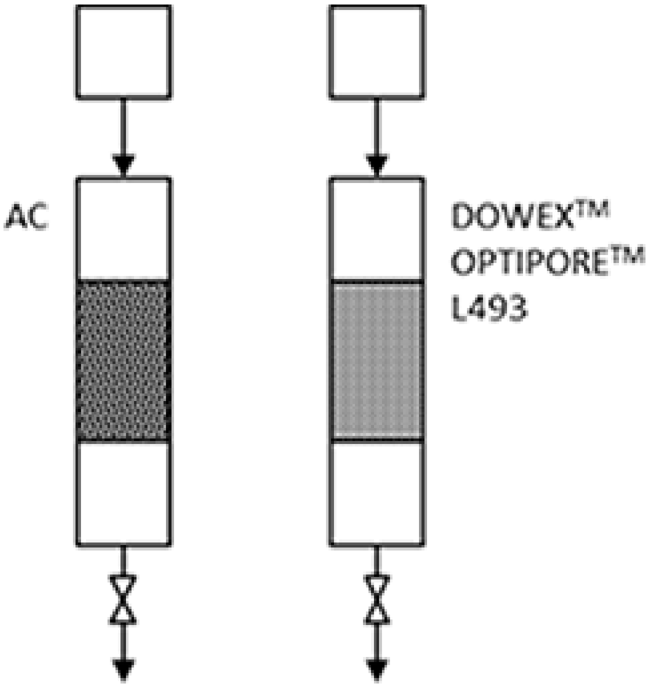

Fig. 2. Experimental set-up.

The method used to quantify the total petroleum hydrocarbons (TPH) is based on a small variation of two official methods: the ASTM D-7066-4 [40] and the EPA 5520-C [41]. These methods are based on the absorbance emitted by the energy bonding between C-H present within the most important organic molecules. Feed and adsorbent-treated water samples are acidified to pH 2 and extracted with tetrachloroethylene. The extract concentration is then determined using Fourier transformed infrared spectroscopy (FTIR). The method generates a spectrum along the region of the aliphatic and aromatic species, in the range between 3060 and 2900 cm^{-1}, where IR adsorption has been calibrated. The area below the spectrum is linearly related to the TPH concentration, expressed as the sum of the individual areas, with a limit of detection (LOD) of 1 mg/l TPH (Fig. 3).

Table 1
Typical physical and chemical properties of adsorbents as specified by the supplier

	DOWEXTM OPTIPORE™ L493	Activated carbon
Matrix	Macroporous Styrene Polymer	Carbon
Physical form	Orange to brown spheres	Black pellets
Particle size diameter	0.3–0.85 mm	1.5 mm
BET surface area	1100 m^2/g	–
Total porosity	1.16 cm^3 /g	–
Average pore diameter	4.6 nm	–
Apparent density	0.62 g/cm^3	2 g/cm^3
Moisture content	50–65%	10%

* "–" Data not available.

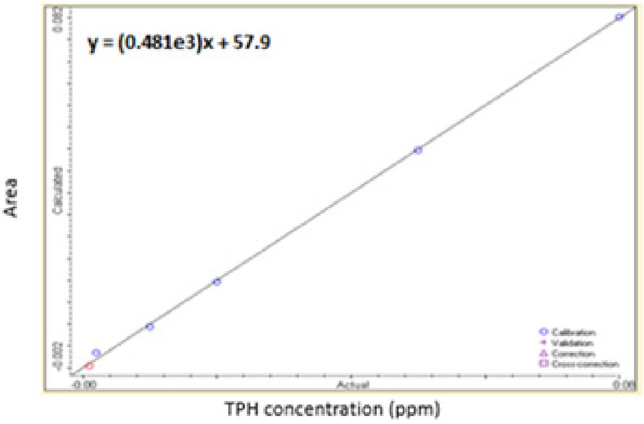

Fig. 3. Calibration curve for TPH determination.

Oil removal has been calculated as the percentage of oil removed from solution after treatment with one of the adsorbents [Eq. (1)]. In the case that concentration in the effluent fell below the LOD, oil removal was calculated as per Eq. (2).

4. Results and discussion

Initial trials showed that treatment with DOWEX™ OPTIPORE™ L493 Polymeric Adsorbent effectively removed the dissolved components of oil, like AC is known to do. In Fig. 3 we can see that there was an important decrease in the FTIR signal generated by sample treatment with both AC and OPTIPORE L493 (Fig. 4).

Operating at a flowrate of 30 BV/h and with an initial concentration of 365 mg/l, both adsorbents are capable of bringing TPH below quantifiable limits, therefore presenting an oil removal capacity of at least 99.9%.

Further experiments also evaluated the performance of AC and DOWEX™ OPTIPORE™ L493 under different flow rate conditions. The capital importance of flow rate when comparing AC and OPTIPORE L493 became evident with a series of experiments with the feed water of 282 mg/L oil content and carried out under different flow rate conditions. Table 2 below shows the TPH concentrations obtained for 15, 30 and 60 BV/h.

We found that at lower flowrates, the oil removal capacity of activated carbon and DOWEX™ OPTIPORE™ L493 polymeric adsorbent was comparable. In both cases, concentration after treatment was below method detection levels. However, as flow rates became larger, the operating adsorption capacity of activated carbon dropped considerably, while the resin was capable of maintaining the same performance. Fig. 5 shows the differences in oil removal between OPTIPORE L493 and AC.

This divergence can be attributed to the fundamental difference of pore structure. AC has a high fraction of micro pores, and therefore is an excellent adsorbent. In fact existent literature indicates that it can have greater adsorption capacity in equilibrium than OPTIPORE L493, as previously indicated. Comparative studies on sorption equilibrium isotherms that have been done with common TPH components such as phenol [30], ethanol and n-butanol [42], all indicate that at equilibrium the adsorption capacity of various activated carbons is higher than OPTIPORE L493 (Fig. 6). However, at large flow rates it is hampered

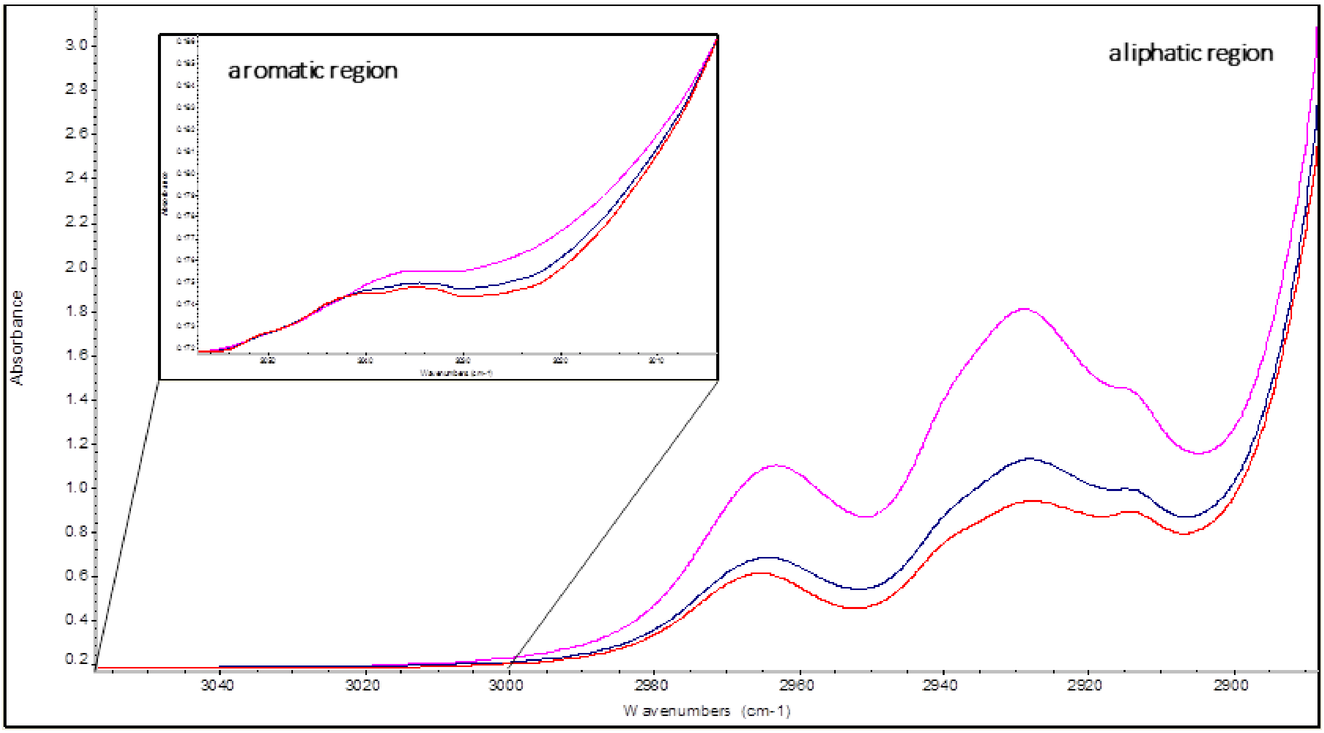

Fig. 4. FTIR spectra for feed (——) (dilution 1:10), and effluent samples treated with AC (——) and DOWEX™ OPTIPORE™ L493 (——) at 30 BV/h.

Table 2
TPH effluent concentration from feed samples of 282 mg/l

	Effluent concentration (mg/l)		
	Flow rate 15 BV/h	Flow rate 30 BV/h	Flow rate 60 BV/h
Activated carbon	< LOD[a]	23	72
DOWEXTM OPTIPORETM L493	< LOD[a]	< LOD[a]	< LOD[a]

a) Samples below limit of detection (LOD) of 1 ppm

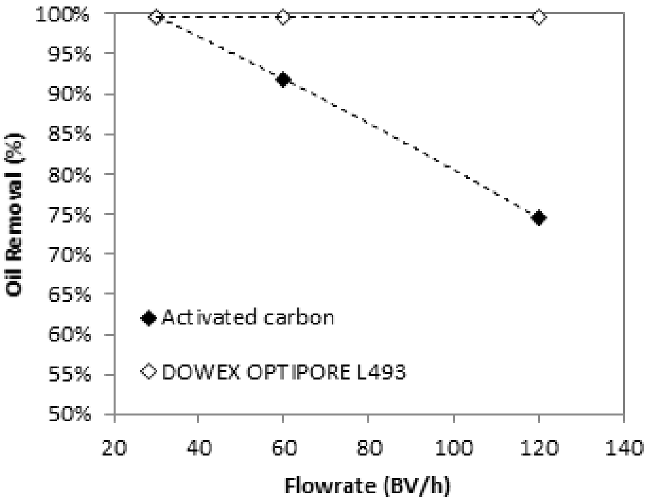

Fig. 5. Oil removal on feed samples of 282 mg/L.

by adsorption kinetics. This is mostly the result of slower diffusion into the pellet micro pores for the adsorption of large molecules – like most petroleum hydrocarbons – due largely to steric effects. The result is that the micro pores in the core region of the activated carbon pellet become unavailable because the contact time is insufficient for the hydrocarbons to reach those sites by diffusion, and only sites close to the surface contribute to the operating adsorption capacity. On the other hand, DOWEX™ OPTIPORE™ L493 has a large macro pore fraction, which effectively acts like a highway down to the more internal micro pores, significantly altering adsorption kinetics. Consequently, at moderate concentrations of TPH, this resin is capable of adsorbing above 99.9% present hydrocarbons up to high flow rate values.

5. Conclusions

Although adsorption in activated carbon is a well-established technology for oil removal, it has certain operational limitations. Findings show that both AC and DOWEX™ OPTIPORE™ L493 polymeric adsorbent are comparable when it comes to effluent water quality. Additionally, both are also capable of removing not only emulsified oil from water, but the more difficult dissolved oil fraction. Furthermore, experiments show that OPTIPORE L493 can still efficiently operate at flow rates where activated carbon experiences a drop in operating adsorption capacity. Such results show that in the long term, the improved operating capacity of the resin represents a need for a smaller volume of adsorbent under such high flow conditions. From a regeneration standpoint, activated carbon is most efficiently regenerated by pyrolysis according to all available literature, whereas OPTIPORE L493 can be efficiently regenerated in-situ with steam. All of this makes OPTIPORE L493 an interesting choice of adsorbent for oil removal.

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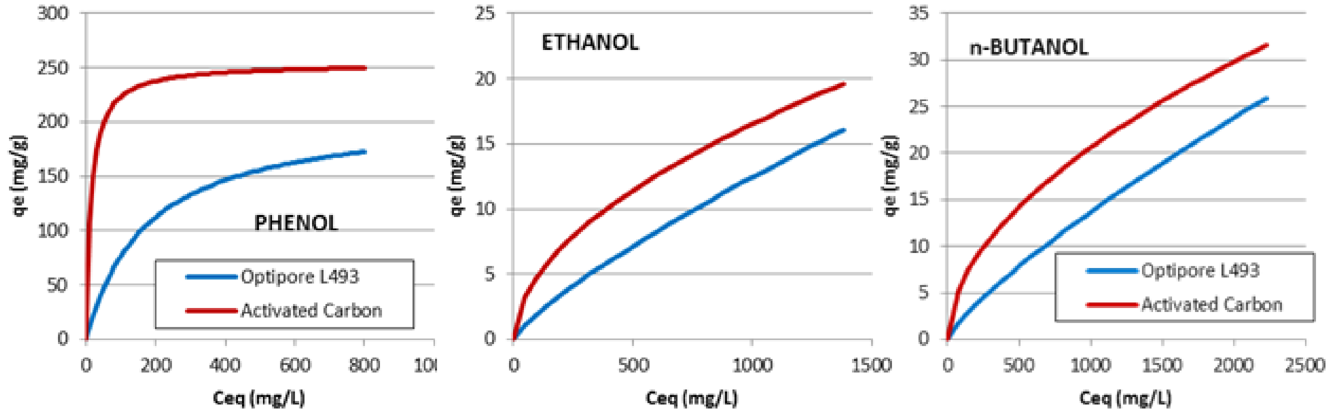

Fig. 6. Sorption equilibrium isotherms for OPTIPORE L493 and AC [31] [43].

Abbreviations

AC — Activated carbon
BOD — Biochemical oxygen demand
BTEX — Benzene/toluene/ethylbenzene and xylene
BV — Bed volume
COD — Chemical oxygen demand
CPI — Corrugated plate interceptors
DW&PS — Dow water and process solutions
FOG — Fat oil and grease
FWKO — Free-water knockout
FTIR — Fourier transformed infrared spectroscopy
GWTC — Global water technology center
LOD — Limit of detection
MPPE — Macroporous polymer extraction
O&G — Oil and grease
OIW — Oil in water
PAH — Polycyclic aromatic hydrocarbons
PW — Produced water
TPH — Total petroleum hydrocarbons
ZLD — Zero-liquid discharge

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