Steam Regeneration of Exhausted Activated Carbon Used in Natural Gas Dehydration by a Triethylene Glycol Unit

B. Djamila[a,*], A. Allali[a,b], B. Youcef[a] and M. H. Sellami[c]

[a]*Department of Process Engineering, Faculty of Technology, University Amar Telidji of Laghouat, BP 37G route of Ghardaïa, Laghouat 03000, Algeria*
[b]*Sonatrach, Oil & Gas Exploration and Services, Rhourde El Baguel, Hassi Messaoud (Ouargla), Algeria*
[c]*Process Engineering Laboratory (PEL), Kasdi Merbah University, Ouargla 30000, Algeria*
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   This study investigated the optimization of the regeneration of exhausted activated carbon (EAC) by hydrocarbons used in natural gas dehydration plants. For this purpose, various methods, including chemical, thermal, and steam regeneration, were used. The experimental results demonstrated the effectiveness of the steam regeneration of EAC at low temperatures, which was further enhanced by the pre-regeneration of EAC with dilute acid (2.5% hydrochloric acid). In addition, the performance of regenerated activated carbon (RAC) against hydrocarbons, prepared from exhausted triethylene glycol (ETEG), was evaluated using the following three parameters: the iodine number in EAC, the refractive index, and the density of TEG regenerated for determining the adsorption capacity. The results showed that the adsorption capacity of RAC on hydrocarbons from ETEG increased with increasing temperature, resulting in highly pure, hydrocarbon-free TEG suitable for use in natural gas TEG dehydration plants. The optimized steam regeneration method with dilute acid pre-treatment showed significant potential for improving the efficiency and sustainability of natural gas dehydration processes. These findings have important implications for the design and operation of steam regeneration units in industrial settings. Moreover, this study contributes to the growing body of research on sustainable and cost-effective methods for hydrocarbon removal in the gas processing industry.

Keywords: Steam regeneration, Exhausted activated carbon, Iodine number, Triethylene glycol, Regeneration efficiency

INTRODUCTION

   Among the most widely used techniques for dehydrating natural gas, absorption with triethylene glycol (TEG) is prevalent. Several natural gas compounds, including benzene, toluene, ethylbenzene, xylene (BTEX), and volatile organic compounds (VOCs), are partially absorbed by TEG in the absorber [1]. Although TEG is regenerated from water in a reboiler, it still contains VOCs that require purification through an activated carbon filter. The regenerated triethylene glycol (RTEG) is then reused for another cycle of natural gas dehydration. TEG units deal with the

environmental aspects related to BTEX and VOCs emissions at the top of the regenerator [2]. Unfortunately, the exhausted activated carbon (EAC) is removed from filters and throughout the entire system due to its saturation with hydrocarbons. The presence of these hydrocarbons is considered a major source of pollution that affects both living organisms and the environment [3]. Moreover, when EAC is exposed to high temperatures, it can cause secondary pollution [4]. Therefore, to prevent the harmful effects of EAC on the environment, various methods, including thermal, chemical, microbiological, and steam regeneration, have been used to regenerate it from adsorbates. Although thermal regeneration is currently the most feasible method used to regenerate EAC, it consumes a lot of energy [5] and causes significant carbon loss [6]. In comparison, chemical

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*Corresponding author. E-mail: brahimidjamila355@gmail.com

regeneration, while having a high adsorption capacity, produces minimal carbon loss and has low energy consumption. However, it results in a lower regeneration efficiency and recovery of the textural properties of activated carbon [7]. Furthermore, the selection of chemicals is restricted to those intended for the treatment of drinking water [8]. Microbiological regeneration takes a long time [8] and is only applied to biodegradable pollutants [9]. Although microbiological regeneration has lower operating costs and is often less toxic to the environment than other methods [5], it is not recommended for water treatment. Steam regeneration follows the selection criteria defined by Pelech *et al.* [10]. During steam regeneration, the high temperature of steam speeds up desorption from the adsorbent. However, the adsorbate is only swept out of the bed when enough steam remains gaseous to sufficiently remove it [11]. Although steam regeneration has been demonstrated to be an effective and economically viable method [12], it has received little attention. Almost all steam regeneration studies have used artificially saturated adsorbents with only one adsorbate. Table 1 provides a summary of previous research on steam regeneration. Pełech *et al.* [10] successfully regenerated 93%

of a mixture of seven components of chloro-derivatives from activated carbon using steam regeneration at 140 °C in a fixed bed. They found that an increase in temperature above 140 °C did not improve the desorption efficiency of 1,2-dichloroethane. Küntzel *et al.* [12] investigated the steam regeneration of zeolite saturated with toluene in a fixed bed reactor at 130 °C. Ying et al. [13] studied steam regeneration of phenol-saturated activated carbon using a rapid small-scale column test and reported that the process yielded optimal results when it was continued for 60 min at 500 °C. In experiments involving the regeneration of dichloromethane from activated carbon beds by hot nitrogen and steam [14], it was found that both methods regenerated 84% of dichloromethane. As part of their study, Schweiger *et al.* [15] investigated the steam regeneration of activated carbon loaded with n-hexane at temperatures ranging from 111 to 127 °C in a fixed bed and using a mathematical model. They took several factors, such as the initial loading of n-hexane, steam flow rate, and flow direction, into account in their study. The regeneration of organic compounds, p-dichlorobenzene, and methyl ethyl ketone from a commercial adsorbent (Ambersorb 563) using steam at 160 °C was

Table 1. Previous Studies on Steam Regeneration

Adsorbate	Regenerator	Adsorbent	Ref.
Toluene	Fixed bed (130 °C)	Zeolites	[12]
Chloro-derivatives	Fixed bed (100-160 °C)	Activated carbon	[10]
Phenol	Small-scale column 400-600 °C	Powdered activated carbon (PAC)	[13]
Dichloromethane	Fixed bed (140, 200 °C)	Activated carbon	[14]
N-Hexane	Fixed bed (111-127 °C)	Activated carbon	[15]
Tetrachloroethylene, p-Dichlorobenzene, o-Chlorobiphenly, Carbon tetrachloride, Methyl ethyl ketone	Fixed bed (160 °C)	F-400 GAC Ambersorb 563	[16]
Methylene blue Phenol	TGA (400-1000 °C)	Activated carbon	[17]
Methylene blue dye	Horizontal fixed bed 970-1000 °C	Activated carbon	[18]
Toluene	Fixed bed (130 °C)	Activated carbon	[19]
Toluene	Pilot scale adsorption column 125, 150 °C	Activated carbon	[20]

successful for six cycles, and no significant degradation was observed in the organic adsorbents [16]. Kato *et al.* [17] investigated the steam regeneration of activated carbon loaded with methylene blue and phenol in the temperature range of 820-980 °C. According to their findings, the regeneration speed increased as the temperature and steam partial pressure increased. Moreover, regeneration efficiency was higher in steam-nitrogen than in nitrogen atmospheres. Xin-hu *et al.* [18] reported on the regeneration of spent coal-based activated carbon in the silicon industry using thermal regeneration techniques and steam as an activating agent. They found optimal regeneration at 983 °C, a regeneration time of 135 min, and a steam flow rate of 2 g min⁻¹. Germerdonk *et al.* [19] conducted experiments using toluene as an adsorbate in activated carbon columns of technical scale with adsorption and steam regeneration in the temperature range of 130-150 °C. It was found that increasing the particle temperature to temperatures far above those of steam condensation significantly reduced the effects of water condensation and adsorption. Using a pilot-scale filter, Gu *et al.* [20] tried to purify indoor air contaminated by toluene using steam regeneration at temperatures between 125 and 150 °C. They found that the more steam used for stripping, the higher the desorption rates. However, the use of a mixture of VOCs as an adsorbate has received little attention in the literature. Therefore, this study aimed at investigating experimentally the feasibility of using steam for the regeneration of EAC with different fractions of hydrocarbons obtained from natural gas dehydration by a TEG unit.

When activated carbon is regenerated, contaminants are transformed into less toxic byproducts and the adsorption capacity of carbon is re-established, extending its lifespan [21]. Activated carbon is conventionally produced by heating lignocellulosic char with steam or carbon dioxide [22]. Compared with CO_2 activation, steam activation produces activated carbons with a narrower pore size distribution, larger surface area, and greater total pore volume [23]. Additionally, steam is readily available and inexpensive in most industrial processes [24] and allows the recovery of the adsorbate without degradation due to the low temperature involved, making it an environmentally friendly method [14]. Moreover, it is easy to separate the organic adsorbate from steam by simple condensation. According to Shah *et al.* [11], steam regeneration is typically conducted at 30-50 °C above the boiling point of VOCs. In this study, decane, with a boiling point of 174 °C, had the largest fraction of hydrocarbons. Following this, steam temperature was set to be in the range of 204-224 °C.

This study focused on the steam regeneration of EAC by hydrocarbons. Environmentally, it is important to avoid secondary pollution with hydrocarbons; economically, it is important to regenerate the EAC and reuse it for its original purpose of adsorbing hydrocarbons from exhausted triethylene glycol (ETEG) in the industry. To improve the industrial application of this adsorbent, an efficient method was developed that consumed less energy and created less ash during regeneration. The regenerated activated carbon (RAC) was tested for its ability to adsorb hydrocarbons from ETEG under different operating conditions.

MATERIALS AND METHODS

RTEG, ETEG, and grains of EAC by hydrocarbons were obtained from a natural gas dehydration unit, which used TEG and was located in Rhourde El Baguel (Hassi Messoud), Algeria, to evaluate the feasibility of the regeneration of EAC through various methods. Also, the RAC was tested for the regeneration of ETEG from hydrocarbons. The principal properties of fresh activated carbon (FAC) are summarized in Table 2. Several methods, including chemical, thermal, and steam regeneration, as well as their combinations, were used to select the most suitable method for the regeneration of EAC.

Table 2. Properties of Fresh Activated Carbon

Particle size (mm)	IN (mg g⁻¹)	Specific area (m² g⁻¹)	Hardness (%)	Bulk density (g cm⁻³)	Ash (%)	Moisture (%)
1.5-1.7	900	850	98	0.45	6	4

Chemical Regeneration

In this method, 1 g of EAC was chemically regenerated by immersing it in 20 ml of 25% hydrochloric acid (HCl) solution in an Erlenmeyer flask and stirring the resultant mixture under magnetic agitation of 900 tr/min for 30 min. Subsequently, the RAC was filtered and washed with hot water until the pH value reached 7.

Thermal Regeneration

The second method was simpler. It involved placing 10 g of EAC into a muffle furnace for 60 min at 950 °C.

Steam Regeneration

In this study, steam regeneration was utilized to treat 1.6 g of EAC within a glass tubular reactor at 200 °C, with steam being introduced through a pump at a volumetric flow rate of 12 ml h^{-1} from the top of the reactor, as shown in Fig. 1. Then, the reaction products, once passed through a condenser, were collected in an Erlenmeyer flask at the bottom of the reactor.

Additionally, combinations of two of the above three methods were examined with the goal of achieving the best outcome at a minimal cost. The chemical method was paired with the thermal method, in which the sample underwent chemical regeneration in a 2.5% HCl solution, followed by thermal regeneration at 650 °C. The chemical method was also paired with the steam method, in which the sample underwent chemical regeneration by immersion in a dilute 2.5% HCl solution, followed by steam regeneration at the lowest temperature (*i.e.*, 200 °C).

Adsorption Test

Following the regeneration of EAC grains, an adsorption test was conducted to determine the adsorption capacity of the RAC toward hydrocarbons from ETEG. Specifically, 1 g of RAC grains was mixed with 10 ml of ETEG, which contained hydrocarbons, in an Erlenmeyer flask and stirred at 900 rpm for 30 min using an electromagnetic stirrer. The mixture was then filtered to obtain the solution beneath the filter and above the anterior chamber. After washing the carbon with hot water and drying it at 105 °C, the iodine number (IN) was measured to determine the degree of its saturation with hydrocarbons. The refractive index and density of RTEG were also measured to evaluate its purity from hydrocarbons and ensure the efficacy of the adsorption.
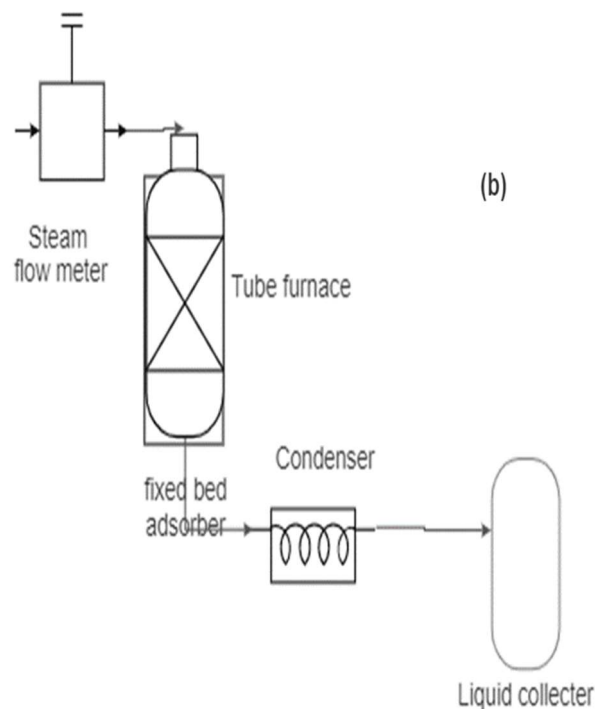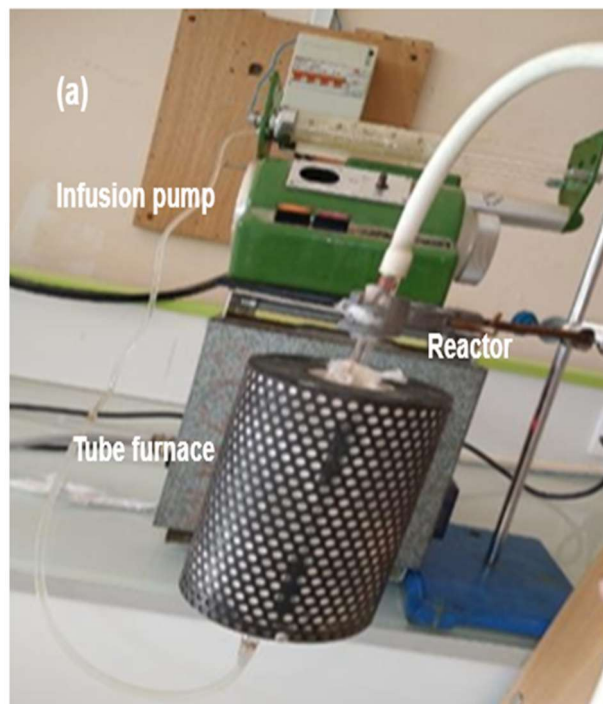

Fig. 1. A photographic caption (a), and a schematic diagram (b) of a fixed-bed steam regeneration experimental setup.

RESULTS AND DISCUSSION

Figure 2 displays the IN of the RAC over time based on different regeneration methods. It is generally observed that the IN of RAC increases as contact time increases, indicating higher regeneration efficiency. Longer regeneration times result in greater dissolution and degradation of organic pollutants, which, in turn, facilitates better mobilization of contaminants from the carbon [25]. These findings are consistent with those of previous studies on thermal [25], steam [17], and chemical [26] regeneration methods. Among the three regeneration methods, thermal regeneration exhibited the highest IN for the RAC, reflecting the highest regeneration efficiency. The chemical method was the second most efficient method in terms of IN, as shown in Fig. 2. Steam regeneration resulted in the lowest IN of RAC and, thus, can be considered the least efficient method. This may be due to the insufficient desorption of the largest fractions of hydrocarbons from the EAC, which was caused by the relatively low temperature (200 °C) used in the steam regeneration process.

The combined use of chemical and steam regeneration methods yielded high regeneration efficiency, at about the same level of efficiency as that of the chemical method alone. Furthermore, the chemical pretreatment of the EAC with 2.5% acid was found to improve thermal regeneration at 650 °C and be effective in removing inorganic compounds blocking the pores of RAC [25].

As depicted in Fig. 3, the high temperature required for thermal regeneration resulted in high energy consumption and produced 4-5% ash content in each cycle. Similarly, Huang et al. reported the same results [27]. When the thermal method was treated at high temperatures, it led to significant loses of porosity and specific surface area, resulting in low regeneration efficiency [28].

Chemical regeneration, however, has its own limitations, such as requiring high solvent concentrations [25], which might deteriorate the structure of the activated carbon [26], and longer regeneration times [4]. Additionally, solvent reuse entails an expensive purification step; thus, it is only recommended for adsorbates that are high-value products [29].

A series of experiments were conducted to examine the effect of temperature and steam flow rates on the efficiency
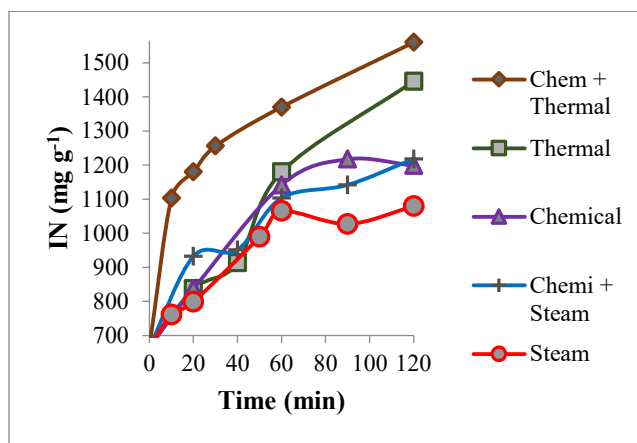

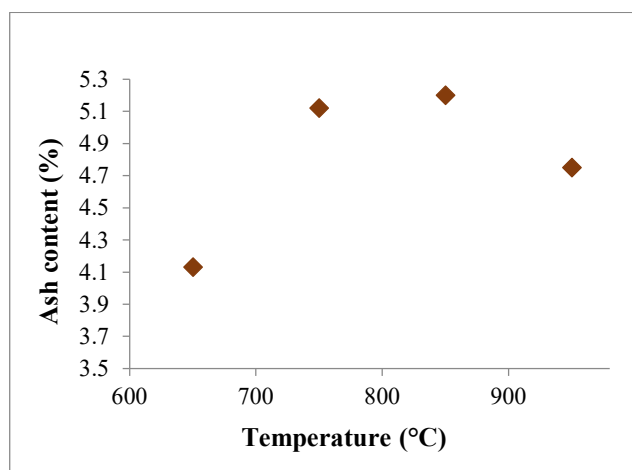**Fig. 2.** The effect of contact time on regeneration efficiency.

Fig. 3. The changes in the ash content following the changes in temperature during thermal regeneration.

of steam regeneration. Figure 4a illustrates the temperature-dependent evolution of the IN of the RAC. The results indicate that higher temperatures led to an increase in the IN values and possible degradation of the adsorbate, resulting in an increased regeneration efficiency [25]. This finding is consistent with those of previous studies that focused on the temperature ranges of 820-980 °C and 970-1000 °C, respectively.

Steam flow rates were increased from 5 to 16 ml h^{-1} to decrease the residence time. Figure 4b shows that increasing the residence time resulted in decreased IN values. This reduction can be attributed to low steam flow rates, which
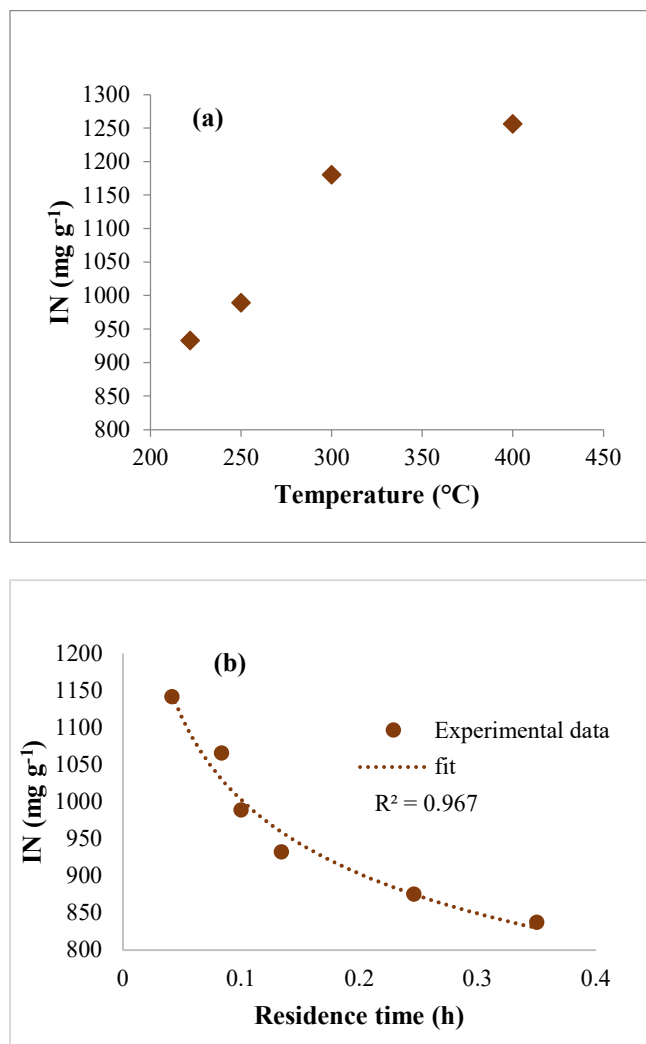
Fig. 4. The evolution of the performance of steam regeneration: The IN as a function of (a) temperature at a fixed residence time of 0.13 h and (b) residence time (at T = 200 °C).

cannot effectively regenerate the adsorbate, leading to lower IN values for the RAC. Similarly, Gu *et al.* [20] observed that a higher amount of steam could lead to higher desorption rates. The increase in the IN and steam flow rates may be due to the adsorbate coming out with the steam [11]. Therefore, using lower steam flow rates is recommended to reduce investment and operating costs since smaller equipment is needed [12]. While Woods *et al.* [30] estimated that steam consumption would range from 3 to 5 kg per kg of desorbed organic compounds, Küntzel *et al.* [12] reported that a

steam/adsorbent ratio of 4 kg kg^{-1} could remove more than 90% of toluene from the adsorber. In this study, a steam/adsorbent ratio of 6 kg kg^{-1} was observed. Hence, this ratio could be reduced to 3.75 kg kg^{-1}, which is equivalent to a steam flow rate of 12 ml h^{-1}. This was achieved by initially treating the EAC with HCl, which represented a lower consumption compared to established norms. Additionally, to achieve an efficient steam regeneration of the EAC by hydrocarbons, it is advisable to preheat the EAC with 2.5% HCl for 30 min at 200 °C and a residence time of 0.13 h, which is equivalent to a steam flow rate of 12 ml h^{-1}.

To assess the adsorption capacity of a RAC using the chemical method followed by a steam method for hydrocarbons from the ETEG, the performance of the RAC was evaluated by measuring the following three parameters: IN in the EAC, refractive index, and the density of RTEG.

Figure 5a demonstrates that the IN of the EAC decreased as the temperature increased until it reached its minimum value, indicating that the RAC had a high adsorption capacity and that the EAC was completely saturated with hydrocarbons at higher temperatures. This increasing trend in hydrocarbon removal efficiency with an increase in the temperature has also been observed in previous studies [31], as in the removal efficiency of different dyes onto sepiolite in a narrow temperature range, including Maxilon Red GRL in the temperature range of 15-45 °C [32], CI reactive blue 221 in the temperature range of 20-50 °C [33], and Maxilon blue GRL in the temperature range of 5-35 °C [34]. These findings suggest that the adsorption of hydrocarbons from ETEG on RAC is an endothermic process. The increase in the temperature may increase hydrocarbon mobility and enable molecules to interact directly with active sites on the surface [34]. The increased temperature may also cause a swelling effect within the internal structure of the activated carbon, allowing heavy hydrocarbons to penetrate deeper [35].

To ensure that the RTEG was pure, the refractive index and density were measured and compared to those of the regenerated and exhausted TEG obtained from the natural gas dehydration unit of Rhourde El Baguel oil field, Ouargla Province, Algeria. As can be seen in Fig. 6, from right to left, (f) is the ETEG saturated by hydrocarbons and appears to be dark as a result of the hydrocarbon content. However, (e) through (b) are TEG regenerated by the RAC at 50, 80, 110,
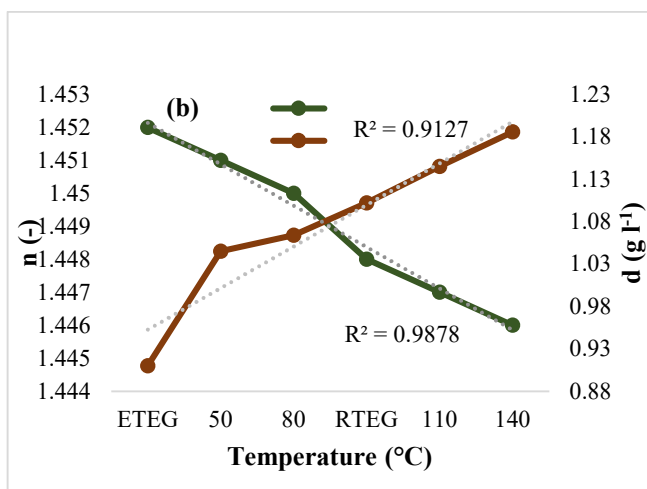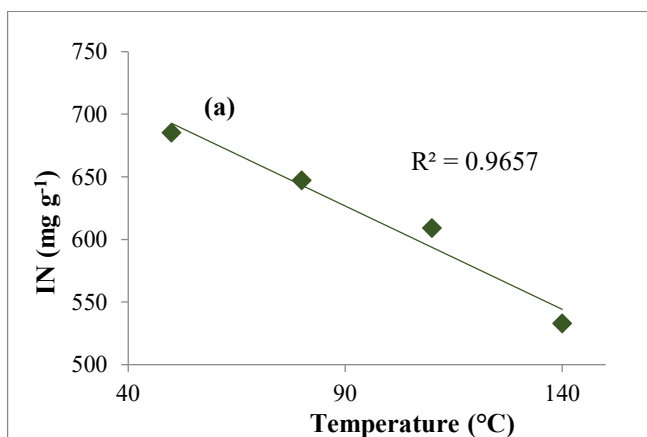
Fig. 6. The temperature-dependent appearance of TEG: (a) the RTEG in the dehydration plant, (b) (c) (d), and (e) the RTEG at 140 °C, 110 °C, 80 °C, and 50 °C, respectively, and (f) ETEG.

Fig. 5. The effect of temperature on the performance of RAC in adsorbing hydrocarbons from the ETEG: The IN as a function of (a) temperature, (b) the density "d" and refractive index "n" of the RTEG as a function of temperature.

and 140 °C, respectively. Moreover, (a) is the RTEG obtained by the dehydration of the natural gas unit and appears to be transparent due to its purity and lack of hydrocarbons.

Figure 5b shows that the refractive index of the RTEG decreased as temperature increased and approached that of the RTEG obtained from the natural gas dehydration unit. The RTEG obtained at temperatures up to 110 °C had a lower refractive index than the RTEG obtained in a dehydration plant, indicating that the TEG was highly pure at high temperatures. Similar to the refractive index, temperature affected the density of the RTEG, as presented in Fig. 5b. The
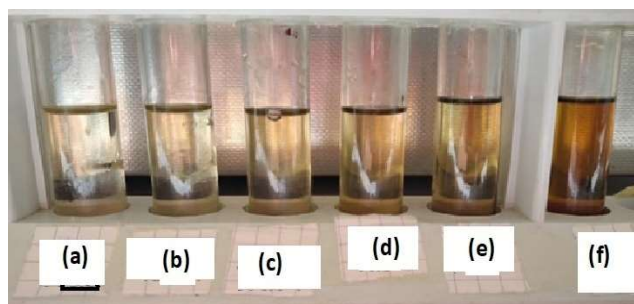
density of RTEG increased as the temperature increased until it approached the density of the RTEG obtained in a dehydration plant. At 110 °C and temperatures above that, the TEG produced had a higher density than the RTEG due to the lack of hydrocarbon traces in the TEG. In conclusion, the results of this study confirm the importance of temperature in improving the adsorption capacity of hydrocarbons from ETEG, which, in turn, can facilitate its regeneration and purification as long as the temperature does not exceed its boiling point.

Numerous experiments were carried out to evaluate the performance of the RAC after multiple adsorption and regeneration cycles. The obtained results are presented in Fig. 7, where it can be observed that the IN of the EAC decreased as the number of cycles increased and settled at a low value of 400 mg g^{-1}, indicating a reduction in its capacity to adsorb hydrocarbons from the ETEG due to aging and loss of its initial adsorption capacity. Similar results were reported by Ying *et al.* [13], who focused on the regeneration of the EAC with phenol using superheated steam and NaOH chemical regeneration, and by Han *et al.* [28], who investigated the regeneration of the EAC with diesel using chemical, ultrasonic, and thermal methods.

Figure 7b presents the properties of RTEG as a function of the number of cycles. It is evident that as the number of cycles increased, the density of the RTEG dropped from its peak and approached the density of the ETEG by hydrocarbons, indicating a decline in the ability of the RAC to adsorb hydrocarbons. The variation in the refractive index of the RTEG with respect to cycle number confirms the above

trend. In addition, it shows an increase in the refractive index, which moved away from that of the RTEG and closer to that of the ETEG with increasing cycles, revealing the negative effect of the number of cycles on the performance of RAC in adsorbing hydrocarbons from the ETEG and, consequently, its purity. Therefore, it is essential to add a small quantity of FAC to the RAC to enhance its adsorption capacity and compensate for production losses in each cycle. As can be seen in Table 2, the IN of FAC was approximately 900 mg g^{-1} and the increase in it compensated for the decrease in the IN of RAC. Furthermore, since the IN of the RAC-FAC mixture follows the simple mixing rule, this approach can be easily applied.

CONCLUSIONS

The experimental results showed that increasing the temperature of steam regeneration from 222 to 400 °C resulted in an increase in the IN of the RAC from 932.71 to 1256.1 mg g^{-1}. However, increasing the residence time from 0.0416 to 0.35 h led to a decrease in the IN of the RAC from 1142.1 to 837.54 mg g^{-1}. Furthermore, a new eco-friendly and cost-effective method for the regeneration of the EAC from hydrocarbons was developed using dilute acid HCl of 2.5% and steam regeneration at 200 °C. It was found that increasing the temperature improved the capacity of RAC to adsorb hydrocarbons from the ETEG. As a result, a highly pure TEG was produced free of hydrocarbons and ready for dehydration of natural gas in a contactor.

Moreover, it was found that an 8-cycle adsorption regeneration reduced the IN of the EAC from 666 to 409 mg/g, directly affecting the purity of the RTEG. After 8 cycles, the density and refractive index of the RTEG (d = 0.928 g l^{-1}, n = 1.46) approached those of the ETEG (d = 0.923 g l^{-1}, n = 1.465), indicating the loss of the RAC adsorption capacity for hydrocarbons from the ETEG. Therefore, it can be stated that it is necessary to consider the number of cycles in designing a steam regeneration unit for an EAC. The results of this study highlight the importance of optimizing the parameters of temperature and residence time to achieve an efficient steam regeneration of the EAC. The results obtained in this study could be used as a useful tool in designing a steam regeneration unit for an EAC.

Nomenclature

BTEX: Benzene, Toluene, Ethylbenzene, and Xylene
d: Density of triethylene glycol
EAC: Exhausted activated carbon
ETEG: Exhausted triethylene glycol
FAC: Fresh activated carbon
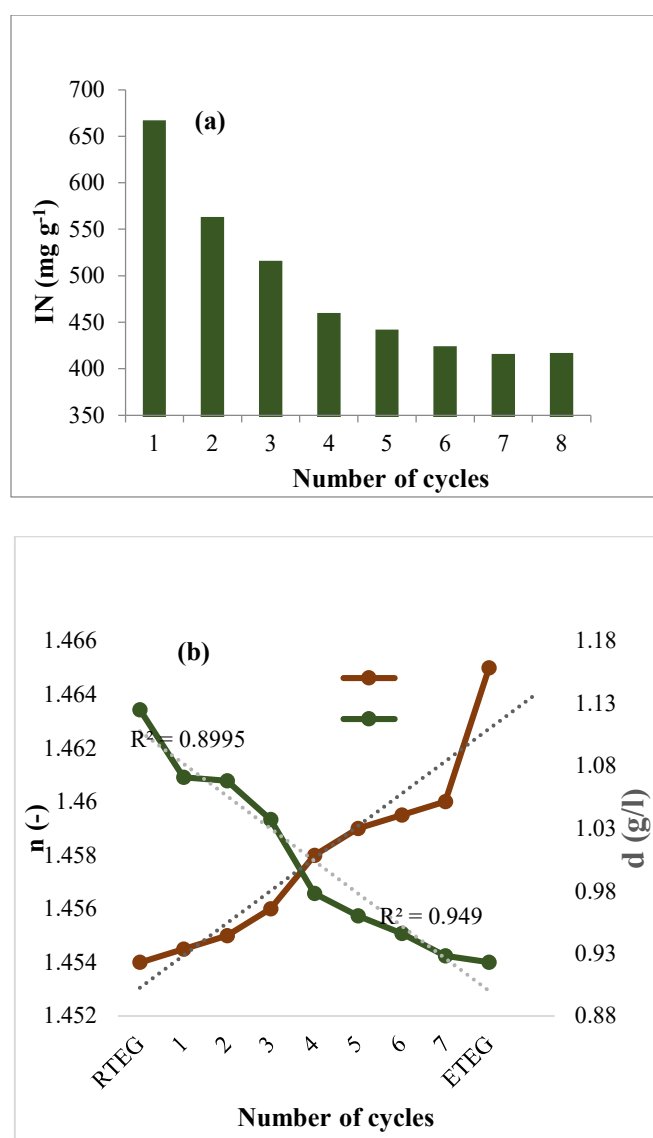**HCl:** Hydrochloric acid
IN: Iodine number

Fig. 7. The evolution of the performance of the RAC in adsorbing hydrocarbons from ETEG with respect to the number of cycles: (a) IN, (b) density "d" and refractive index "n" of the RTEG.

n: Refractive index
RAC: Regenerated activated carbon
RTEG: Regenerated triethylene glycol
TEG: Triethylene glycol
VOCs: Volatile organic compounds

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