Application of chitosan absorbent in reducing the amount of airport wastes containing ethylene glycol pollution

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Abstract
In this research, chitin and chitosan were used to remove the water pollutants and wastewater through adsorption process, due to their distinctive biological and physical-chemical characteristics, and their efficiency was investigated to remove ethylene glycol cationic pollutant from aqueous environments of wastewaters generated from airport. Using the ethylene glycol leads to the formation of wastes containing EG[1] mixed with mineral salts. There are several methods for treating these wastes, in the research; we intend to investigate the efficiency of the adsorption phenomenon to remove ethylene glycol from industrial wastes. Non-continuous absorption experiments were conducted to examine the effect of the factors affecting the removal process, including the effect of contact time of absorbent with pollutant, PH, initial concentration of the pollutant and the amount of consumed absorbent. The results indicate that chitosan is suitable for absorbing ethylene glycol from industrial wastewaters, and by increasing absorbent weight, the absorption rate also increases.

Keywords: adsorption, airport wastewater, chitin, chitosan, ethylene glycol

INTRODUCTION

Water pollutants such as toxic metals and organic compounds has caused serious environmental problems, and therefore, it is necessary to control their presence in the environment. Among all the proposed methods, using the green methods is an economic need. The importance of chitin and chitosan increased recently, on one hand due to the fact that they represent renewable and biodegradable resources, and, on the other hand, due to their functionality at applications in such fields as biology, pharmacy, bio-technology, medicine and the chemistry of materials (Islam et al. 2011). The chitin ($C_8H_{13}NO_5)_n$ forms the exoskeleton of insects or of other arthropods; it can also be found in lichens or some species of fungus. The chitin can be described as cellulose for which a group of hydroxyl in each monomer replaced with a group of acetyl-amine, this increases the hydrogen bonds between the molecules involved in the bond, which is a factor in increasing the resistance of chitin membranes (Kurita 2006). From a chemical point of view, chitin is poly N acetyl D glucosamine β-(1,4) -2-Acetamido-2-dezoxi-D-glucoză or N-acetyl-D-glucosamine β- (1,4) N-acetyl-D-glucosamine (Azam et al. 2014). Chitin, which was

identified in 1811, is the second most abundant natural polymer after cellulose (Altaner et al. 2014).

Since the purpose of the study is to remove ethylene glycol from water resources, we firstly review the research conducted on the pollutant in recent years. The airport wastes are normally mixed with the wastes generated in the buildings of airports and its treatment method becomes possible by biological processes (Adeola et al. 2006, Liu and such. 2007, Gabrieli 1997). Orecki et al. have extensively studied the recycling of WEG[2] and its application as raw materials in various chemical industries (Orecki et al. 2006). In recent years, given the problem of a shortage of energy and greenhouse gas emission, more effort is clearly needed on renewable energies, even from wastes. The anaerobic process which can produce methane gas and less nutrients with high loading rate, has been investigated (Blika et al. 2009, Diamantis and Aivasidis 2010, Vlassis 2012). In 2005, Kennedy used an anaerobic sludge reactor (UASB) to purify WEG, and the

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[1] Ethylene Glycol
[2] Wastewater containing diluted ethylene glycol

efficiency of removal rate was reported to be 93% (Kennedy and Barriault 2005).

The process of airport de-icing operation, which consists of two major components, the deicing and anti-icing, is performed on aviation industry in the cold seasons. The annual consumption of this solution is 300 ton, of which 80% is ethylene glycol. De-icing and anti-icing, which are used in airports, with the main percentage of various concentrations of ethylene glycol, is a chemical product with environmental effects. Since 1992, extensive international researches have conducted on the wastewater collection and recycling of ethylene glycol. Using the ethylene glycol leads to the formation of wastes containing EG[3] mixed with mineral salts. There are several methods for treating these wastes, which also include the destruction of toxic organic matter. These methods include Ozonation and filtration, using electric current.

In the research, we intend to investigate the efficiency of the adsorption phenomenon to remove ethylene glycol from industrial wastes. So far, no operations have been undertaken to reduce the pollution load by the proposed method. Since the material uses not only in airport de-icing solution, but also in railroad industries, even runways and roads, it has been working more on the process of its collecting and recycling, but due to lack of infrastructure in the country, its collection for recycling is not possible. In Iran, no research has been conducted on the de-icing wastewater.

EXPERIMENTAL

Materials

The materials used in this research include: ethylene glycol (molecular formula $C_2H_6O_2$), with molecular mass of 62.07 g.mol^{-1}, density of 1.11 g.cm^{-3} and 99% purity which was selected because it is a pollutant which is partially soluble in water, and purchased from Merck Company. Industrial zeolite of chitosan was used as absorbents. Standard solutions of 0.1 M HCl and 0.1 M NaOH were used to adjust the pH of the solution.

Equipment

In the research, after conducting the necessary steps to absorb pollutants by absorbents used to measure the removal rate of pollutant from aqueous solution, 2200/3800 GC-Ms made by Varian Company (AUS) was used. But, in order to get better results, TOC[4]-31A made in Japan was used to carry out the experiments. In order to increase the contact surface of the absorbent with the solution containing pollutant, a stirrer L-81 made by a German company, Hudolth, was used to mix the solution. After completing the desired time, a centrifuge (Model *H-108 N*, Kokusan, Japan) was used to isolate

suspended solid absorbent particles from the solution. Digital PH meter was used to adjust the solution PH.

Using a double beam UV–VIS spectrophotometer, UV spectrum of the chitin extract was recorded in acetonitrile solution. The studied solution was inserted in quartz with an optical path of 1 cm.

General Techniques

This research was carried out in the laboratory of airport service companies. For initial and periodical qualitative experiments, type I de-icing materials and type II, III and VI anti-icing materials were designed and it has the necessary facilities to carry out experiments on these materials according to the following standards.

Type I (de-ice): MIL-A-8243, AMS 1424

Type II, III, IV (anti-ice): AMS 1428

Pour point of these materials was measured at -70 °C according to ASTM-D97 standard[5] and viscosity of these fluids was measured using viscometer. The corrosion experiment on metals is measurable according to the method specified in MIL-A-8243 standard. PH was measured according to ASTEM-D-1287 standard and the density was measured using a picnometer and precision scale. Fully automatic digital refrac*tometer* can directly determine the concentration of these materials in percent as well as their freezing point. All laboratory equipment is calibrated using Certified Reference Materials (CRMs) to ensure the accuracy and precision of the experiments. Refractometer shows the concentration of ethylene glycol and the freezing point of the diluted solution.

Preparation of Chitosan Absorbent

The chitin is of nitrogen derivate with a structure resembling to the structure of cellulose; it is insoluble in water and resistant to acids, bases or a large number of organic solvents. In the case of crustaceans, the chitin is impregnated with calcium carbonate that shows a higher asperity (Rahman and Halfar 2014). Due to the insolubility, the chitin hasn't a large applicability and therefore, it enters into a partial deacetylation process, thus the chitosan is obtained (Ehrlich et al. 2013). Chitosan is a product which is obtained after the removal of acyl. The chitosan is natural polymer with properties such as bio-compatibility and bio-absorbability. It is non-toxic and soluble in weak organic acids. Chitosan has several commercial and bio-medical applications. It can be used in agriculture for the treatment of seeds and as a bio-fertilizer, protecting the plants against fungus infection; when wine production, it is used to prevent the alteration of wine (Berger et al. 2014). On industrial scale, chitosan is used in the process of water filtration. Following the process of obtaining the chitosan through the deacetylation of chitin (which usually is not complete), the studies related to the analytical properties of chitin and chitosan, and the studies concerning the

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[3] Ethylene Glycol
[4] Total Organic Carbon

[5] American society for testing and materials
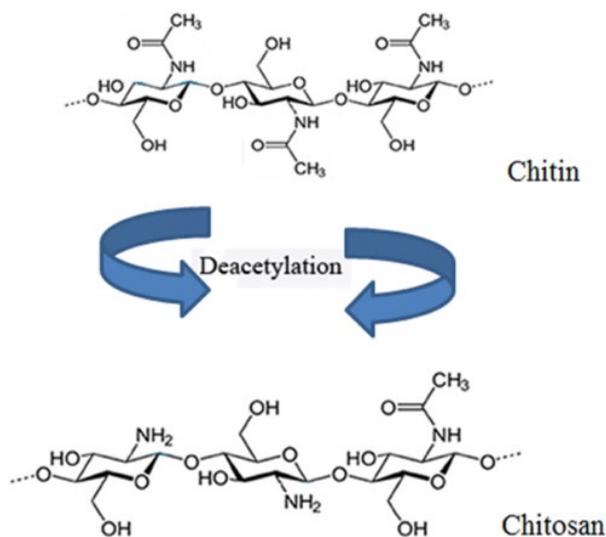
Fig. 1. Chitosan Deacetylation

degree of deacetylation performed to produce chitosan were considered to be important. In order to obtain the natural biomass quantities having the characteristics of biocompatibility and bio- absorbability, these researches can be important (Islam et al. 2017, Komi et al. 2016, Usman et al. 2016).

In this research, fresh tiger shrimps' shells were used. The shells were thoroughly washed with water and soaked in a 0.5% NaOH for 4 h to remove shrimp residues from the skin. The shells were again washed with water and dried for 2 h in an oven at 60ºC and then turned into powder with milling machine. Then, the extraction of chitin from the shells was carried out based on the method proposed by Chang et al. (1997) that is as follows.

This operation was performed using a normal caustic soda solution at 90ºC for 2 h. The weight ratio of shrimp shell powder to caustic soda solution was 1 to 20. The shell residue was then filtered, and in order to reach neutral PH, the remaining material washed on a filter with distilled water. The residues from the prior steps were placed in a 1.4 normal HCl solution for 1 h. The weight ratio of shell to acid was 1:10. The shell residue was then filtered and the remaining material washed on a filter with distilled water until the PH was neutral. The resulting chitin was yellow and must be colorless. To produce chitin free of carotenoid pigments, chitin was washed with acetone until its color is clear and white. In this step, the chitosan was produced by chitin deacetylation (**Fig. 1**). Deacetylation was carried out at 100ºC for 6 h in a concentrated soda solution (50%). Suspended materials were then filtered in soda solution and the aforementioned material (chitosan) which remained on the filter were washed using distilled water to reach neutral PH. The chitosan was then dried in an oven at 60°C for 1 h (Younes and Rinaudo n.d., Zargar et al. n.d.).

The unmodified chitosan can be dissolved only in acid solution because of the intermolecular hydrogen bonds, which limits its application as hydrogel (Baxter et al. 1992). The chitosan shows a crystalline structure, which its polymorphism depends on its physical status. Its varied structures contain an anhydrous form, a hydrated form and several salts. The chitosan represents an alternative for obtaining synthetic polycations that used as flocculation agents. The water treatment offers possibilities ranging from removing the Humic acid from potable water to waste treatment techniques or mud dehydration. Since chitosan has the characteristic of oxygen barrier, the chitosan films could be used in food or pharmaceutical products packaging. Chitosan shows an anti-microbial activity against a range of microorganisms (fungus, yeasts, and bacteria). The anti-microbial properties of the chitosan are among its advantages compared to other biomaterials with possible applications in the packaging industry.

RESULT AND DISCUSSION

Absorption Studies

Using a double beam UV–VIS spectrophotometer, UV spectrum of the chitin extract was recorded in acetonitrile solution. The studied solution was inserted in quartz with an optical path of 1 cm. **Fig. 2** shows the absorption spectrum of the acetonitrile solution of chitin extract. The infrared spectrum was obtained by samples included in potassium bromide tablets. The amount of sample included in one tablet (diameter of 13 mm) is around 1 mg, and the amount of potassium bromide (having the role of binding agent) is 200 mg. The sample containing mixture and the potassium bromide is homogenized until the fine granulation is formed and a homogenous consistency is achieved. Then, in a hydraulic press, the powdered material undergoes a pressure. Before the pressure is applied and while pressing, the inside of the matrix is vacuumed to avoid generating the air micro-bubbles in the tablet. The presence of these micro-bubbles, on one hand, may disturb the optical behavior of the tablet, and on the other hand, it can create the micro- fractures when the pressure is removed. Therefore, a compact and transparent mass is obtained, which is homogenous from an optical point of view (Kjaergaard et al. 2014).
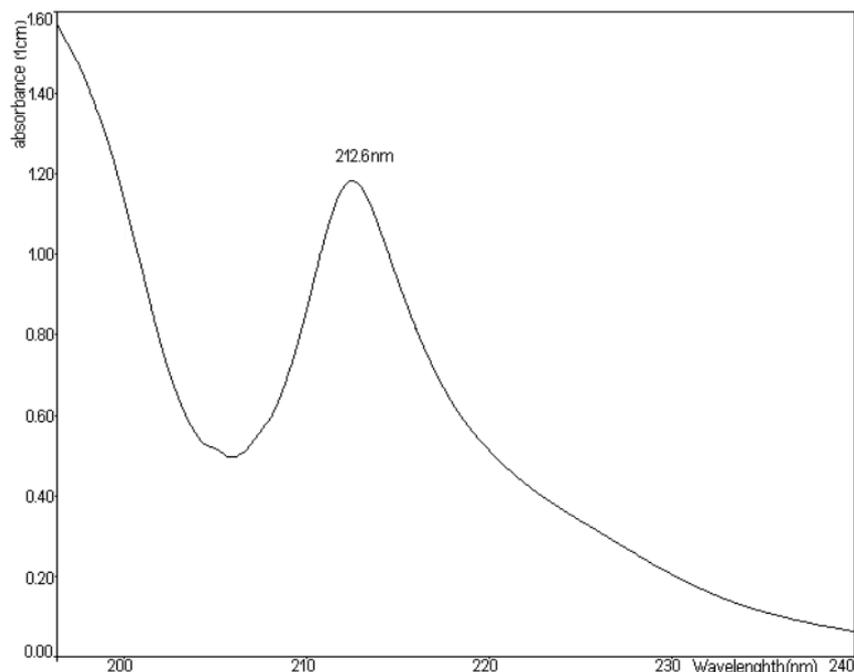
**Fig. 2.** The Absorption Spectrum of Acetonitrile Solution of Chitin Extract
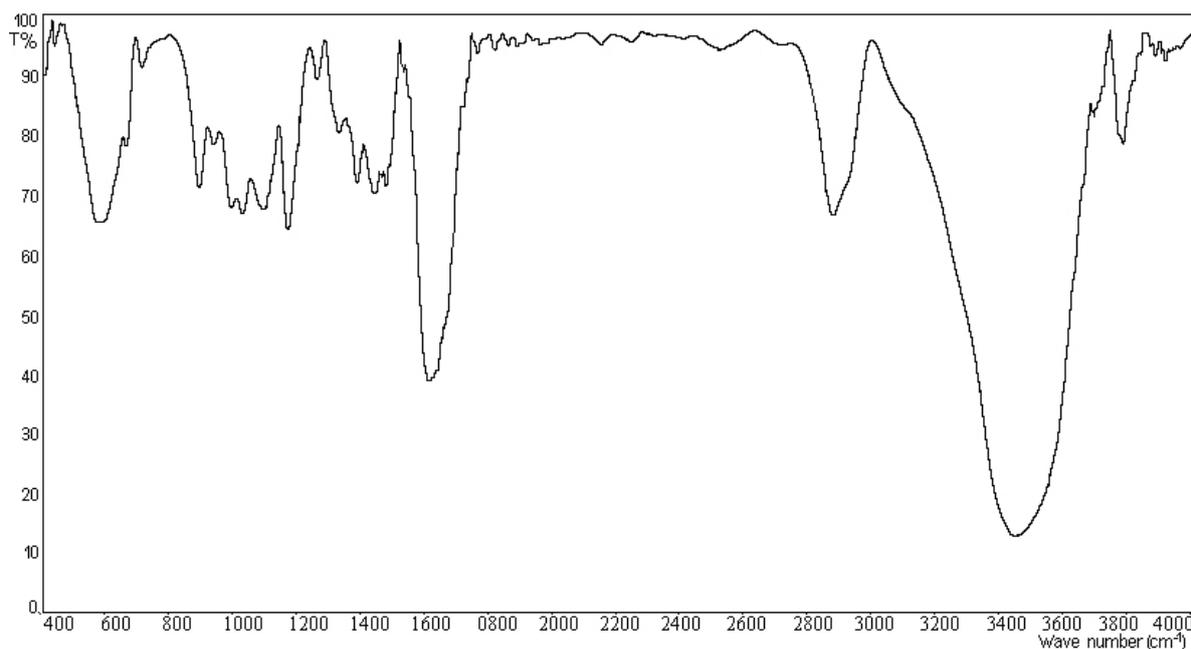
Fig. 3. Infrared Spectrum of Chitin Extract in the Field of 400-4000 Cm^{-1}

According to the above-mentioned reasons, the analysis of infrared spectrums was started. **Figs. 3** and **4** represent the infrared spectrum of the chitin in the fields of 400 – 4000 cm–1 and 400–2000 cm–1, in terms of transmittance vs. wave number (υs –symmetric stretch; υas – asymmetric stretch; δ–deformation; ω–wagging). The bands are generally large due to the macromolecular structure of the compound and the numerous intermolecular bindings of hydrogen, manifested even in the solid state of the sample.

Ultraviolet Spectrophotometry

Although the molecular structure of the chitin, which is predominantly saturated, does not allow a substantial absorption (it is not an efficient chromophore), the spectrum shows absorption bands between 200-260 nm, and a maximum absorption that is 212.6 nm (Kjaergaard et al. 2014, Lee et al. 2012). This band can be caused by electronic transitions of n–π* type, which is produced by the type II amid of chitin. But even these
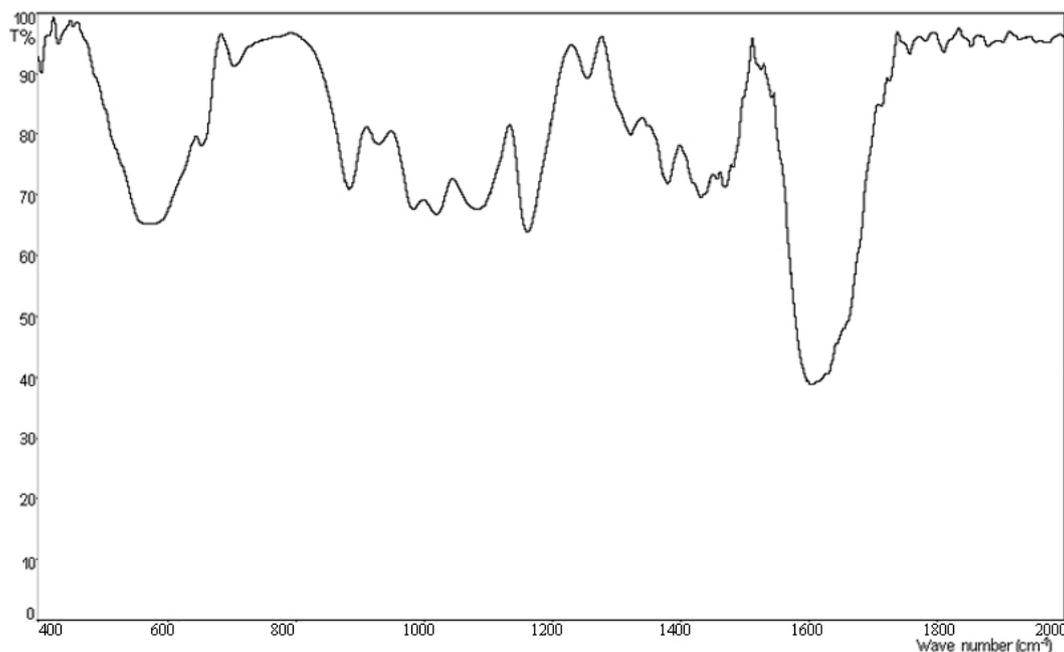
Fig. 4. Infrared Spectrum of Chitin Extract in Field of 400-2000 Cm^{-1}

Table 1. The preparation of standard mixtures

m1 (mg Chitin)	m2 (mg Chitosan)	Acetylation Degree (%)	Total Intensity
10.3	989.7	3.999 ≈ 4	15.8044
10.4	191.2	8.004 ≈ 8	31.1149
18.6	181.5	12.016 ≈ 12	43.4620
13.4	86.6	15.998 ≈ 16	58.2787
70.1	329.8	20.003 ≈ 20	76.5525

signals are low, not being able to provide an appropriate sensitivity.

In the ranges of 3280 cm^{-1} (chitosan) and 3275 cm^{-1} (chitosan oligomers), the overlapping of (OH) on υs (N–H) is visible. In the range of 2870 cm^{-1}, the vibration of υ (–C=O) of the amide group CONHR is observed in chitosan. The bands of 1568 cm^{-1} (chitosan) and 3285 cm^{-1} (chitosan oligomers) are related to (–C=O) of the amide group, and δ (NH$_3$). Absorption in the ranges of 1417 cm^{-1} (chitosan) and 1419cm^{-1} (chitosan oligomers) is related to δ (OH). Absorption in the ranges of 1372cm^{-1} (chitosan) and 1375cm^{-1} (chitosan oligomers) is related to δ (–CH$_3$). Absorption in the ranges of 1152 cm^{-1} (chitosan) and 1148 cm^{-1} (chitosan oligomers) is formed by υas (C=O) oxygen bridges resulting from the deacetylation of the chitosan. The bands 1060 cm^{-1} (chitosan) and 1080 cm^{-1} (chitosan oligomers) are related to υ (C=O) by the bindings of C–O–H, C–O–C and CH$_2$CO. The bands of 890cm^{-1}(chitosan) and 890 cm^{-1}(chitosan oligomers) are related to the polysaccharide structure which is determined by ω (C-H).

Absorption in the range related to amide I is due to the vibration of C=O in the amidic group. According to the fact that the absorption is associated with the groups of acetyl of the molecule, it is used for stating the deacetylation degree of chitosan sample (the more

advanced is the acetylation degree, the more intense is the band). In other words, the Absorption in the range of 3450 nm is the internal standard. Five mixtures were prepared in different proportions of chitin (acetylation degree of 100%) and chitosan (acetylation degree of 3 %) for the calibration of the connection between the total intensity of amide I band and the acetylation degree [28]. Table 1 shows the data from the preparation method of the standard mixtures. The degree of acetylation of the obtained mixture is calculated by the formula 1.

$$acetylatio\ n\ degree\ (\%) = \frac{100 \cdot m_1 + 3 \cdot m_2}{m_1 + m_2} \quad (1)$$

Where: m_1 is chitin mass (in mg), m_2 is chitosan mass (in mg).

Fig. 5 shows the normalized overlapping images for amide I absorbance band in the standard mixtures. **Table 1** represents numbers corresponding to the individual spectrums related to the acetylation degree.

Preparation of Standard Solutions

Using standard methods, the solution containing ethylene glycol pollutant at a concentration of 1000 ppm was prepared in distilled water. Standard ethylene glycol solutions with different concentrations in distilled water were prepared by dilution of mother solution in the concentration range of 400-1000 ppm.
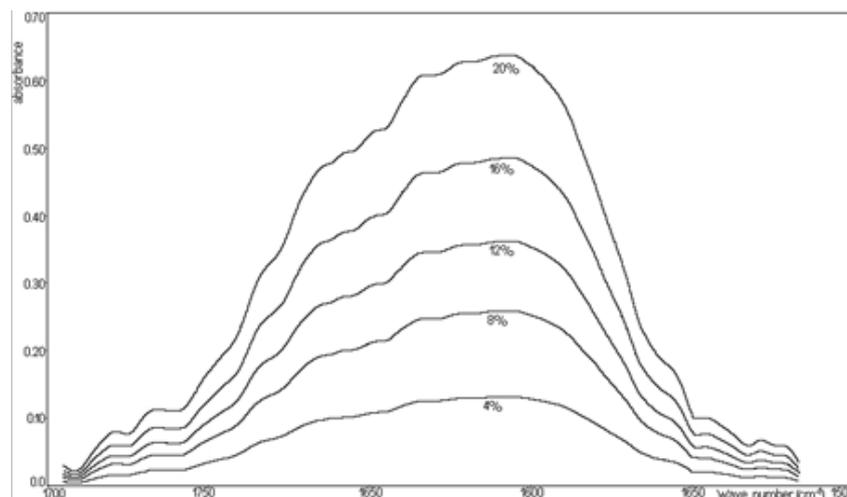
Fig. 5. The normalized Overlapping Images of Amide I Absorbance in the Standard Mixtures
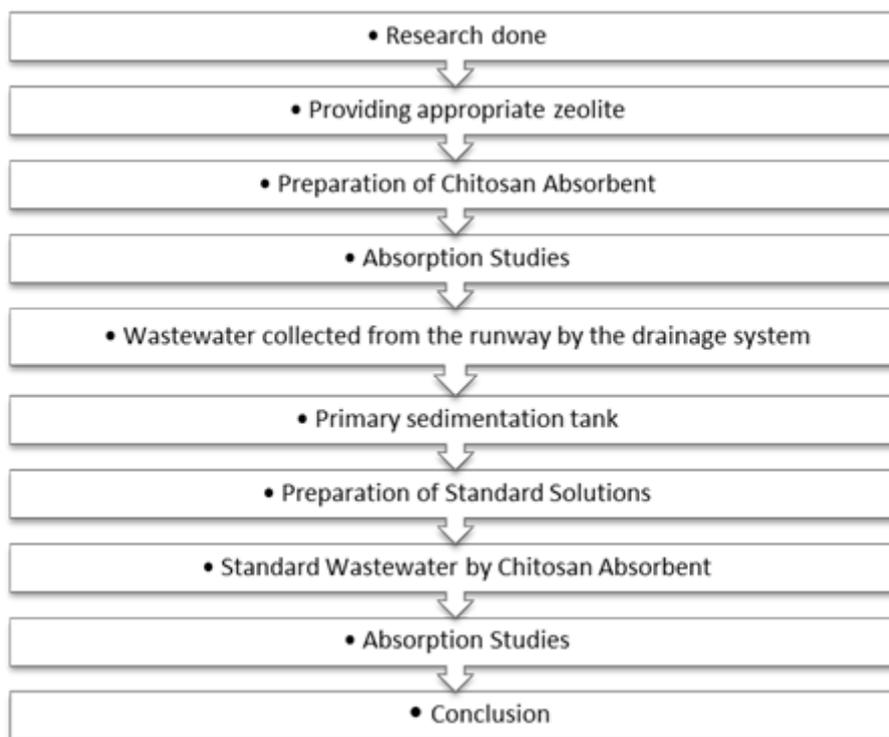

Fig. 6. The research process carried out

Airport Waste Treatment (Glycol Recycling)

Polyhydroxy compounds such as glycols and glycerol are difficult and expensive to recover from aqueous solution because of their high boiling points and affinity towards water. Propylene glycol (1,2 - propanediol, PG) and ethylene glycol (1,2-ethanediol, EG) are commodity chemicals produced via hydration of their respective epoxides in aqueous solution. In order to obtain pure PG and EG from aqueous solution, water must be boiled off; the energy utilized in this process constitutes a substantial portion of the cost of recovery. Vacuum distillation is often used to lower temperatures and thus avoid formation of undesired polyglycols and decomposition products and to reduce energy cost.

Recycling systems are emphasized on standard separation techniques to remove water and suspended solids and some surfactants, corrosion inhibitors and other additives from wastes. The glycol recycling process generally involves several steps, which include filtration, ion exchange, nanofiltration, flocculation, reverse osmosis, and distillation. The process depends on the system used and the desired result (for example, the recycling product will be used in the aviation industry or the automotive industry). Biological Recycling is
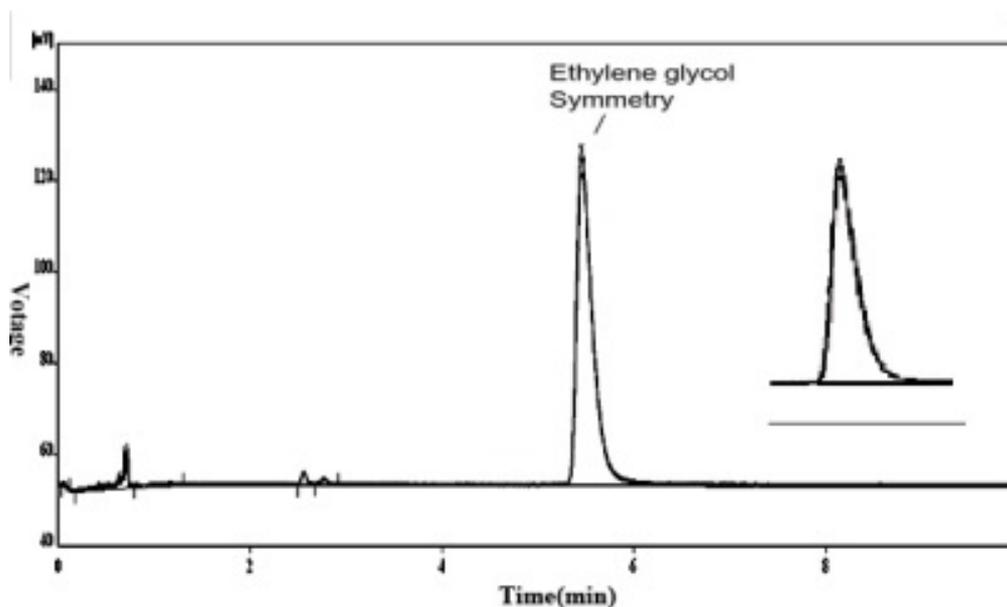
Fig. 7. Electrocardiogram Prepared for 0.05% Ethylene Glycol

another method for recycling that requires different performance and many time.

Recycling glycol from the airport waste reduces the pollution level that comes to surface water and land. The technologies are available for recycling fluids containing at least 5% glycol. Recycling the glycol reduces the amount and impact of waste and reduces the waste disposal charges. In addition, recycled glycol may be sold. The value of recycled glycol depends on the type of glycol and its concentration and purity. In the research, the collected fluid was first mechanically filtered by ultrafiltration, in which impurities such as sand and gravel, grass, oil, concentrator used and other "inappropriate materials" were removed. The next step in the chemical purification process is accomplished through the unit of absorption and ion, anion and cation exchanger. The purpose is to remove heavy metals, chloride and so on. The PH of the fluid is also controlled during this process. These steps lead a water-glycol mixture, which still contains excess water. The research cycle workflow shown in Figure 6 is shown.

Experimental Method

The study of absorption of ethylene glycol by chitosan absorbent was performed taking into account the impact of different parameters such as absorbent amount, contact time, initial concentration of ethylene glycol solution, PH values and absorption rate.

To do so, in each experiment, using a calibrated cylinder, 50CC of the solution of ethylene glycol at a predetermined concentration of pollutant was poured into a series of erlenmeyer flask, and then different quantities of absorbent were added in the range from 0.1 to 2 g. The solution was rapidly stirred at the speed of 200 rpm and the absorption rate was measured at

different time intervals. At the end of the determined time, a portion of the solution was isolated, centrifuged and examined for absorption studies. In order to investigate the effect of the above factors on absorption, each of these factors were investigated at different ranges.

To do so, the contact time between 30 and 120 min, absorbent amount of 0.1-2 g, PH range of 2-8, initial concentration of ethylene glycol between 400 and 1000 ppm for all experiments were investigated. The residual ethylene glycol absorption in the solution was then measured by TOC and the percentage of removal was calculated by the formula 2:

$$Percentage\ of\ removal\ rate\ of\ ethyleneglycol\ from\ the\ sample\ solution$$
$$= \frac{Initial\ concentration - final\ concentration}{Initial\ concentration} \quad (2)$$

Measuring the removal rate of pollutant from aqueous solution was performed using GC device. In the electrocardiogram prepared for 0.05% ethylene glycol (**Fig. 7**). The analysis was conducted for several times.

Given that aqueous samples, when using, are converted to vapor phase and increased in volume, for example, each macroliter of water produces about 1,400 macroliter of vapor, which causes the some portion of vapor to not enter the column, and goes to different parts through the injector that is out of the glass liner. Another problem is polar columns. In the research, given that the material identified is ethylene glycol and our selected column is Innovax column, it is necessary to mention this point that these columns themselves have glycol base, i.e., in fact, they are polymeric glycols and inherently has a lower repeatability than other polar columns and are not able to provide the appropriate sensitivity.

Standard Wastewater by Chitosan Absorbent

Due to the proper interaction between chitosan absorbent in the design and the ethylene glycol pollutant present in the wastewater, by increasing in concentration, there were still sites to absorb the pollutant, and therefore, the absorption rate of pollutant increased on the absorbent. In another parameter, which is related to the amount of absorbent, as expected, by increasing the amount of absorbent, absorption increased, but after increasing the amount of absorbent to a certain extent, absorption didn't increase anymore. As shown in the graph, the increase in absorption had an ascending trend. As shown in Figure 8, increase in the contact time was not a significant parameter, and since the absorbent was suitable absorbent with high efficiency, absorption took place within the first minutes and before the first 30 min, and the absorption of ethylene glycol pollutant occurred via the solution.

CONCLUSION

The effect of the effectiveness of adsorption on the removal of ethylene glycol from the industrial wastewaters by low cost and biodegradable absorbents like chitosan was performed. For this purpose, zeolite absorbents were used and their effectiveness was investigated to remove the cationic pollutant of ethylene glycol from the aqueous environments of the contaminated wastes of the airport in different conditions in a discontinuous manner. The discontinuous absorption tests were carried out to investigate the influence of effective factors, including contact time of the absorbent and the pollutant, pH, initial concentration of the pollutant and the amount of adsorbent consumed. Considering the experimental design, chitosan absorbent has a high potential for removal of ethylene glycol from aqueous solutions and facilitates the absorption of the pollutant, and thus leading to the highest removal of the ethylene glycol from aqueous solutions.

ABBREVIATIONS

EG   Ethylene glycol
WEG   Wastewater containing diluted ethylene glycol
TOC   Total organic carbon
ASTM   American society for testing and materials

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