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ANILINE BIO-ADSORPTION FROM AQUEOUS SOLUTIONS USING DRIED ACTIVATED SLUDGE

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ABSTRACT

Aniline is among aromatic hydrocarbon contaminants which is highly used in chemical industries and different processes and have many adverse impacts on the creatures and the environment. Thus, in this study efficiency of nano-photocatalytic process of zinc oxide nanoparticles and ultraviolet ray in removal of aniline from synthetic wastewater is investigated. The dried sewage sludge, which was activated using potassium carbonate (K_2CO_3) at 600 °C, was used as a sorbent in this study. All experiments of Aniline adsorption were carried out on activated ash produced in a batch reactor using 100 mL Erlenmeyer with effect of changing parameters of pH 2 to 11, absorbent dosage of 1- 10 g/L and 50-250 mg/L aniline by spectrophotometer colorimetric method during contact time of 120 min. In addition, Freundlich and Langmuir isotherms on optimum data were determined. Results showed that efficiency of about 0.99 % was obtained during 120 min, absorbent dosage of 10 g/L and pH 6 in presence of 50 mg/L aniline. Also results indicated that data follow second order kinetic and Langmuir isotherm (R^2 0.99).

KEY WORDS: Aniline, sludge, adsorbent, potassium carbonate, absorbed, bio-absorption

INTRODUCTION

The number of organic compounds produced within the new century has exceeded half a million and about 10,000 new compounds are added each year (Nemerow *et al.*, 2014; Saeidi *et al.*, 2017). Impacts of volatile organic compounds and volatile toxic organic compounds have been found recently in collection networks and sewage treatment units and it has caused concerns (Biglari *et al.*, 2017; Biglari *et al.*, 2016a). Thus using new methods with capability of decomposition of these compounds seems necessary (Khosravi *et al.*, 2017; Rahdar *et al.*, 2017).

These organic compounds especially aromatic hydrocarbons are common contaminants that are

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widely used in many industries such as painting, plastics, textiles and petrochemical industries (Zhang *et al.,* 2009; Rahdar *et al.,* 2016; Sajjadi *et al.,* 2016). Among aromatic hydrocarbons, aniline is highly used in chemical industries (Dong *et al.,* 2007; Karimaei *et al.,* 2017). Aniline is among chemical compounds which is resistant in the environment and their presence in surface waters and wastewater of sewage treatment units has created concern (Vieno *et al.,* 2006). Aniline or Aminobenzene is an organic compound with formula $C_6H_5NH_2$ which consists of a benzene ring attached to an amino group. Aniline is fatty and colorless and it is easily oxidized in the air and forms a sample in red-brown color. Like most volatile amines, it possesses the odor of rotten fish. Aniline is toxic, it is easily igniting, burning with a smoky flame (Al-Johani and Salam, 2011). Aniline as a toxic substance, it has a water solubility of 3.5%, so this solubility in water not only increases risk of its presence in the sewage system, but also it can be found in drinkable water sources as a toxic chemical (Wang *et al.,* 2007). In general, solubility of organic or chemical compounds beyond the standard limits is a threat to the health of living organisms (Biglari *et al.,* 2016b). Some important aniline derivatives include phenylenediamine and diphenolamine, which are used as oxidants. Methylene diphenyldiisocyanate is the most commonly used aniline (Liu *et al.,* 2012). Aniline is a chemical carcinogenic compound that causes cancerous tumors in animals and increases the risk of bladder cancer in humans (Wu *et al.,* 2012). Aniline has a high toxicity and is rapidly absorbed through the skin and, if swallowed or inhaled, causes hemoglobin to be converted to methemoglobin and cyanoside (Jin *et al.*, 2012). Long and continuous exposure to Aniline can reduce appetite and weight, anemia, effects on the nervous system, and damage to kidney, liver and bone marrow (Wu *et al.,* 2012). Health rules have determined limits for Aniline exposure as 2 ppm for 8 hours for skin absorption and 5 ppm for 8 hours for air contact (Bhunia *et al.*, 2003; Agency, 1999, Ruder *et al.,* 1992). On the other hand, this compound produce rapid reactions in blood causing hemoglobin conversion to methemoglobin (Mieyal and Blumer, 1976). Aniline is widely used in chemical industries as precursor as well as in painting factors, rubber, pharmaceutical production, plastics and pesticides (Brillas and Casado, 2002; Qi *et al.,* 2002). Over 150 types of compounds are derived from Aniline. In USA, 62 types of industrial units use Aniline and/or produce it (Brillas and Casado, 2002). According to the US Environmental Protection Agency, the maximum concentration of aniline in groundwater is 6 μg/L (Goncharuk *et al.,* 2002). Aniline production in the United States and China is about 80,000 and 457,000 tons annually, respectively (Qi *et al.,* 2002). In Iran, about 156 tons of this combination are imported annually and used in various industries such as - petrochemicals, rubber and painting (MOUSAVI *et al.,* 2011; Taherkhani *et al.,* 2015). Hence given this amount of consumption, its presence in the wastewater of these industries would be high, and considering creating various environmental problems, necessity for appropriate removal of these organic compounds from aquatic environments is critical. Several different physico-chemical methods have been considered for removing aniline and aromatic compounds from aqueous solutions including chemical oxidation, sequestration, ion exchange, electrochemical methods, irradiation, activated carbon and volatile ash (Li *et al.,* 2017; Bláha *et al.,* 2017; Pirsaheb *et al.,* 2017). Using natural adsorbents such as biodegradable activated sludge is the other efficient and cheap process for removal of organic compounds (Tang *et al.,* 2017). Such adsorbents are generally activated using heat or chemicals and are known as activated carbon. Since these materials have high adsorption capacity they are widely used as absorbents especially for removal of organic contaminants from aquatic environments (Liu *et al.,* 2017). The ability for absorbing activated carbon related to high level of surface depends on the volume and size of pores. In addition, activated carbon adsorption depends on the way of activation and source of activated carbon (Singh *et al.,* 2017). Activated carbon is widely used for removal of chlorine, separation of gases and purification of contaminated air, recycling of heavy metals and food industries, and removal of potentially polluting compounds such as aniline in aqueous solutions, and considering importance of economic issues, researchers have always sought for cheaper alternatives (Essandoh *et al.,* 2017; Alipour *et al.,* 2017; Khaksefidi *et al.,* 2016; Narooie *et al.,* 2016). Using biodegradable activated sludge as an inexpensive absorbent for removal of organic compounds is also recommended in some studies (Tsai *et al.,* 2008). Therefore, this study was conducted to evaluate the efficiency of the bio-adsorption process of activated dried sludge in removal of aniline from aqueous samples.

MATERIALS AND METHODS

All chemicals used in this study, except sludge, were prepared from Merck Co., Germany. In order to conduct this fundamental – applied research work, which was done in a batch system on a laboratory scale, firstly required amounts of wet sewage sludge were prepared from a wastewater treatment plant in Torbat Heydarieh, Mashhad, Iran, which was designed and operated by active sludge method. The sludge was washed and rinsed several times with distilled water. After washing, the sludge was dried using the oven at 105 ± 3 °C for 60min and then it was mixed with a weight ratio of 50% by weight of K_2CO_3 to produce activated sludge slag, and placed in a furnace at 600 Centigrade degrees for 2 hours for conversion into ashes, and kept cool in the desiccators until they were used (Okman et al., 2014). Following preparation of the samples, efficiency of activated dried adsorbent process in water samples containing Aniline with concentrations of 50 – 250 mg/l was examined with effect of parameters as initial pH 2 to 11, absorbent dose of 1- 10 g/l during 120 min of contact time. In addition, the process kinetic and absorbent adherence to Langmuir and Freundlich adsorption isotherms was also investigated. The variation of aniline concentration was studied based on the standard method of water and wastewater experiments by colorimetric method using Jenway spectrophotometer Genova Plus model (Federation and Association, 2005). During the test, mixture of adsorbent and water was stirred by Jar Test apparatus, VELP SCIENTFICA JLT6 model, with a speed of 40 rpm to prevent deposition. The pH of the samples was adjusted by using normal hydrochloric acid and sodium hydroxide 1 and 0.1 using a pH meter of the Ultera Basic UB-10 model manufactured in the United States. At the end of each process, the samples were made filtrated using standard Whatman No: 40 µm filter paper, made in the United Kingdom, and were tested for aniline concentration determination.

RESULTS AND DISCUSSION

Effect of pH

Absorption of Aniline from aquatic environment totally depends on the solution's pH, because pH influences electric charge of the absorbent surface and changes the degree of aniline ionization. Figure 1 indicates results of pH changes on the adsorption process. Investigation of this figure indicates that efficiency of absorbed Aniline reduces by increasing pH. In this study, optimum pH, which gives highest removal efficiency, was obtained as 6. Change in pH affects adsorption process during decomposition and division of dependent groups, at active adsorbent surfaces. Therefore, it leads to change in kinetic of adsorption reaction and equilibrium properties of adsorbent and absorbent material in the adsorption process. The adsorption of various anionic and cationic species on such adsorbents is defined by adsorption competition of H^+ ions and OH^- ions with absorbed material. Aniline adsorption decreases with increasing pH of the environment. Aniline is a weak base which its adsorption is decreased in the competition with the OH^- acidic medium at high pH (Biglari *et al.*, 2016a; Jin *et al.*, 2012). Results of this study showed that pHpzc, as one of the main parameters of adsorption process for specifying adsorption charge point, was 6.7 in this work and thus pHs smeller than this value has positive charge and it is a reason for efficient adsorption of Aniline as a weak base material in not much acidic pHs (Khorramfar *et al.*, 2009). Aniline, which is a weak base, reacts with H^+ ion in acidic pH, and turns into anilinium ion with positive charge (Bayramoglu *et al.*, 2009). Since pKa of anilinium is 9.37, efficiency of contaminant removal is reduced at highly acidic conditions. These findings are consistent with findings by Han et al. which reported efficiency of Aniline adsorption reduces at highly acidic and basic pHs (Han *et al.*, 2006).

Impact of Two Absorbents

Figure 2 indicates effect of absorbent dose of 1- 10 g activated dried sludge ash per liter of sample on efficiency of adsorption at aniline initial concentration of 10 mg/L and pH 6 during 120 min. Investigation of Figure 2 indicates efficiency of aniline removal increases by increasing initial absorbent dose. Highest removal efficiency was obtained at dose 10 g/L as 99 percent. Increasing active surface and pores on ash surface increases active surface of contaminant adsorption, thus efficiency increases. In fact, aniline absorbed at absorbent mass unit reduces and these findings are consistent with findings by Gadeer *et al.* (Qadeer and Rehan, 2002). Senturk *et al.* maintain that with increasing absorbent dose, unsaturated residual adsorption sites reduce adsorption capacity, in

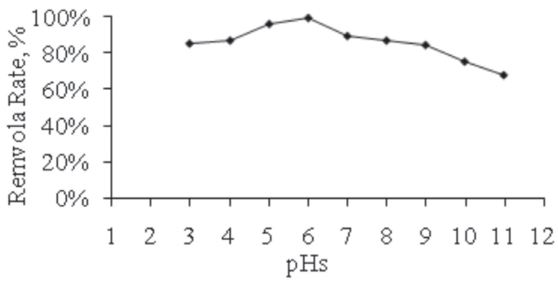

Fig. 1. Effect of pH
(Aniline 50 mg/L and adsorbent dosage 10 g/L)

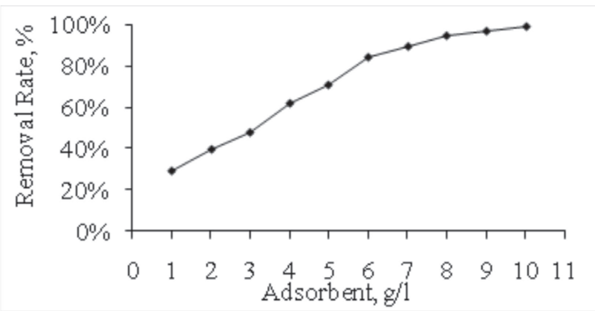

Fig 2. Effect of adsorbent dosage
(Aniline 50 mg/L and pH 6)

addition, adsorbent particles at high levels reduce surface area and increase length of the propagation pathway (Senturk *et al.,* 2009).

Effect of Contact Time

In order to investigate effect of contact time on efficiency of aniline removal elimination process, samples with initial concentration of 50 mg/L with adsorbent 10 g/L were prepared. Effect of time on aniline adsorption process is shown in Fig 3. Investigation of figure 3 shows that highest efficiency of contaminant removal was obtained at time of 120 min. Also Figure 3 indicates speed of adsorption is higher at initial reaction times. This

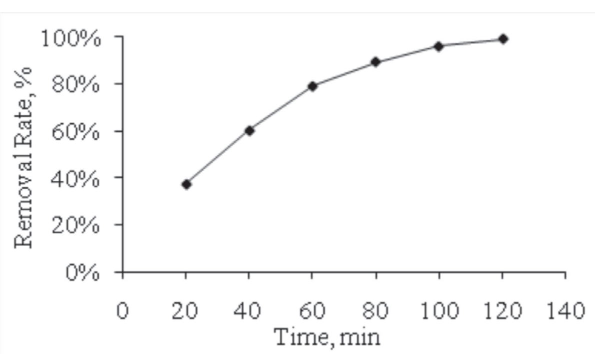

Fig. 3. The effect of contact time
(Aniline 50 mg/L, pH 6 and adsorbent dosage 10 g/L)

increased adsorption is due to higher number of collision between contaminants and adsorbent. The longer is retention time, probability of collision is also increased and the absorption of the contaminant by the desired adsorbent increases, and then the removal process goes slowly (Srivastava *et al.,* 2006).

Effect of Aniline Concentration

Figure 4 indicates equilibrium capacity of adsorbent at different initial aniline concentrations. Investigation of Figure 4 indicates that adsorption capacity increases with increasing initial concentration of aniline, while aniline removal efficiency shows inverse trend at the same time. Increasing aniline concentration increases stimulus of mass transfer, and therefore, accelerates the passage of aniline molecules from solution to the adsorbent-surrounding liquid layer and eventually towards the surface of adsorbent particles, and the action increases the absorption capacity. While reduction in removal efficiency was due to the limited number of active sites on adsorbents at high concentrations, which were saturated with aniline, and this decrease tendency for adsorption sites of removal efficiency. In other words, at lower concentrations, access of aniline molecules to adsorption sites is higher than higher concentrations (Qadeer and Rehan, 2002).

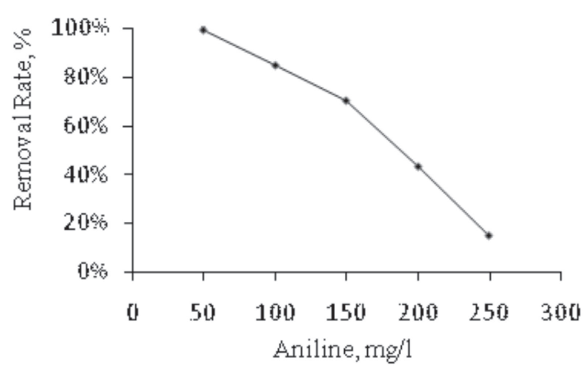

Fig. 4. Effect of initial aniline concentration
(Time 120 min, pH 6 and adsorbent dosage 10 g/L)

Isotherm and adsorption kinetics

There are different models for explaining data obtained from adsorption experiments at scientific sources, main of which is Langmuir and Freundlich adsorption isotherms. Adsorption isotherm analysis is done in order to develop an equation for accurate representation of results and for purposes of system

Table 1. Kinetics and adsorption isotherms (25 °C)

in-particle penetration			pseudo-second-order			pseudo-first-order		
R^2	c	k_{pi}	R^2	q_e	K_2	R^2	q_e	K_1
0.59	10.11	0.086	0.99	9.89	0.63	0.68	1.94	0.076
Langmuir				Freundlich				
q_0		K_l		R_l	R^2	K_f	n	R^2
10.42		0.038		0.28	0.99	0.04	0.51	0.97

design. Thus two models are used for expressing relationship between absorbed phenyl and its equilibrium concentration at the used solution. Table 1 show the data of active dried sludge adsorption in aniline removal from Langmuir and Freundlich adsorption isotherms. As shown in Table 1, correlation coefficient in the model for activated dried sludge absorbent is high. The speed constant of the pseudo-first-order, pseudo-second-order equation, and in-particle penetration is shown in Table 1. In this model, correlation coefficient of Aniline pseudo-first-order equation and in-particle penetration was low, which suggests low correlation of this material. Thus, three kinetic models of pseudo-first-order, pseudo-second-order and in-participle penetration had good correlation on adsorbent for Aniline. Since RL values result in zero to one in Langmuir model, therefore phenyl adsorption on sludge adsorbent is on the basis of multilayer and heterogeneous adsorption of absorbed material on the absorbent. Freundlich isotherm, unlike Langmuir model, is based on multilayer and heterogeneous adsorption of absorbent material on the absorbent. Freundlich constant (n) is smaller than 1, thus Freundlich isotherm model is optimal mathematically and in terms of intensity of absorption. So considering regression coefficients obtained in both isotherm models, it is observed that data obtained from adsorption process are better described with Langmuir and Freundlich model. According to findings in this work it can be concluded that activated dried sludge adsorbent is an inexpensive and accessible material which has appropriate potential for Aniline removal like other adsorbents. Also given correlation coefficients it can be concluded that equilibrium data of adsorption process on modified adsorbent for Aniline mostly adhere to Langmuir isotherm (R^2 = 0.99) more than Freundlich isotherm, so it can be said that adsorption of Aniline by activated dried adsorbent is as single-layered and homogenous. Al-Johani et al. reported that adsorption of Aniline on multi-walled carbon nanotubes better adhere to Langmuir isotherm (Al-Johani and Salam, 2011).

CONCLUSION

Results of this study are shown that process efficiency was optimum at not much acidic pHs and well adhere to Langmuir isotherm. Results showed that efficiency of about 0.99 percent was obtained during 120 minutes with absorbent dose of 10 g/l and pH 6 in presence of 50 mg/l Aniline. Also results indicated that data follow second order kinetic and Langmuir isotherm (R^2 0.99). So, activated dried sludge adsorbent has good capability in adsorption of aniline from aquatic environments. Thus, this adsorbent can be used as an inexpensive and efficient adsorbent for Aniline removal from aquatic samples.

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