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Comparative study of influence of molecular structure of hydrogenocarbonate contaminants in adsorption process, using cactus pear forage (*Opuntia fícus*) as biomass

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ABSTRACT
This work used adsorption technique to evaluate adsorption efficiency of an easily accessible biomass in Paraiba, cactus pear forage (Opuntia fícus), to remove organic contaminants from water/lubricating oil and water/diesel oil systems present in water bodies. Study was developed to obtain kinetic curves to characterization of adsorption dynamics of lubricating oil and diesel oil in contact with biomass. For this, methodology described by Lima et al. (2014), which used mandacaru biomass (Cereus jamacaruna) in particulate form to remove gasoline/diesel mixture, which consisted of: preparing biomass on particulate form, for further granulometric analysis, using sieves; conducting experiments to investigate adsorption kinetics and to obtain adsorption isotherms to complementation of equilibrium study. In despite low adsorption values confirmed by kinetics and equilibrium adsorption graphs, at some points 50% of adsorbed lubricating oil was obtained, which may characterize cactus pear forage as a relatively efficient adsorbent. Low values obtained on adsorption of diesel oil may have been influenced by molecular structure of diesel, constituted of long chains that may have favored oscillations on adsorption by means of cactus pear forage biomass.

Keywords: Adsorption, Cactus pear forage, Diesel oil, Lubricating oil.

1 INTRODUCTION

In general, Brazil is a privileged country in terms of volume of water resources, as it has 13.7% of world's fresh water. However, availability of these resources is not uniform since more than 73% of fresh water available in the country is in Amazon basin, which is inhabited by less than 5% of population. Only 27% of Brazilian water resources are available to other regions, where lives 95% of country's population (ÁGUA..., 2017).

Water can have its quality affected by most diverse activities of man, whether domestic, commercial or industrial. Each of these activities generates characteristic pollutants that have a certain implication in the quality of receiving body (PEREIRA, 2017).

Concept of polluted water includes not only changes in physical, chemical and biological properties of water, but also addition of liquid, solid or gaseous substances capable of rendering water unfit for different intended uses (A POLUIÇÃO..., 2017).

Among most used processes to decontamination of aquatic bodies, adsorption has been highlighted as an efficient method and adaptable to the most different applications.

Adsorption operation is widely used in industry, mainly to purification of drinking water, treatment of contaminated effluents with low concentrations of organic contaminants, recovery of solvents, among other applications (BRINQUES, 2005). Adsorption success as a separation process depends on the choice of adsorbent material and optimization of process variables (LIMA, 2010).

Materials employed in adsorption of organic pollutants are diverse and can be organic, synthetic organic and organic from animal or plant origin. Materials of plant origin come in a variety of forms, including straw, hay, corn cob, peanut bark, bark fibers, cotton fibers and cellulose of cotton-like plants (RIBEIRO *et al.*, 2000).

Among varieties found in the Paraíba semi-arid region, cactus pear forage (*Opuntia ficus*) is presented as an alternative biomass to using as adsorbent of hydrocarbons in water bodies.

1.1 LITERATURE REVIEW

1.1.1 Water pollution

Urban stormwater carries significant quantities of debris and pollutants that include litter, oils, heavy metals, sediment, nutrients, organic matter and micro-organisms, and has been recognized as one of the major sources of diffuse pollution to aquatic environment (DAVIES**;** BAVOR, 2000).

Increasing advance of industrial technology has resulted in generation of hazardous wastewater which, if launched into the public sewage system, without proper pretreatment, affects its structural integrity by containing corrosive, flammable and explosive pollutants (SOUZA; LIMA; SILVA, 2006).

Subsurface migration, fertilizer runoff from farms, storm water from streets, and other forms of diffuse pollution are now recognized as having serious environmental consequences. In more than 50 percent of estimated cases in which water quality goals are not achieved, diffuse pollution is cited as the main cause (NOVOTNY, 1994).

Oil is at same time one of most important sources of energy and environmental pollution. Being composed of complex mixtures of hydrocarbons, of various molecular weights and structures that ranging from a light gas (methane) to a heavy solid, consists of hydrogen and carbon, which are prevailing elements, including up to 98% of some crude oils and 100% of many refined products (ALEIXO; TACHIBANA; CASAGRANDE, 2007).

1.1.2 Hydrocarbons

Petroleum hydrocarbons, basic designation of which derives from structural arrangement of carbon and hydrogen atoms of which are composed, cause great damage when in the aquatic environment (ALEIXO; TACHIBANA; CASAGRANDE, 2007).

Some of hydrocarbons that contribute most to pollution of aquifers are those that make up lubricating oil and diesel oil.

Lubricating is a fluid that, applied to machines, creates an impermeable layer between its parts, reduces heating and prevents that parts in movement cause friction or wear. It is a soft and easily deformable material that has weak secondary bonds (LIMA, 2009).

Oil can seep into the soil, contaminating it and, when it reaches underground groundwater, also pollutes well and fountains water. If it is discharged into wastewater drainage networks, it pollutes water receiving means, as well as causing damage to treatment stations (GUERRA *et al.*, 2012).

Energy source, diesel oil is a fuel derived from petroleum, and its composition contains paraffinic, oleopheneic and aromatic hydrocarbons, mainly formed by atoms of carbon, hydrogen and, in low concentrations, sulfur, nitrogen, oxygen, metals, among others (FERREIRA, 2008).

Diesel oil is used in internal combustion engines and compression ignition (OLÉO DIESEL, 2016).

Generally, diesel engines operate with excess of oxygen, obtaining reductions in hydrocarbons and carbon monoxide emissions; however, among main pollutants emitted by engines, most prominent are particulate materials, considered biggest polluters in large urban centers. Diesel oil has a grid of emitters of pollutants to environment, being main factors being cetane number, sulfur content, aromatics content and density (FERREIRA, 2008).

1.1.3 Adsorption

Removal of organic products in environment has been a major technological challenge because, innumerable times, conventional treatment technologies are not able to do it efficiently. For this reason searches for effective technologies to remove them, with a low cost has grown (SOUZA; LIMA; SILVA, 2006).

Adsorption process using biomass as adsorbent has been a potentially attractive and economical alternative to purification and separation in petroleum, food, fine chemicals and biotechnology areas.

It is a valid option to removal of diluted pollutants in liquid effluents, as well as to recovery components of high added value diluted in industrial streams (SCHEER *et al.*, 2002).

Adsorption is based on principle that a solid surface in contact with a fluid tends to accumulate a surface layer of solute molecules due to imbalance of existing surface forces (RUTHVEN, 1984). Adsorption phenomenon is closely related to surface tension of solutions and its intensity depends on temperature, nature and concentration of adsorbed substance (adsorbate), nature and aggregation state of adsorbent (finely divided solid) and fluid in contact with adsorbent. Fluid, surface and components retained by surface are main elements of adsorption (CAMPOS *et al.*, 2012).

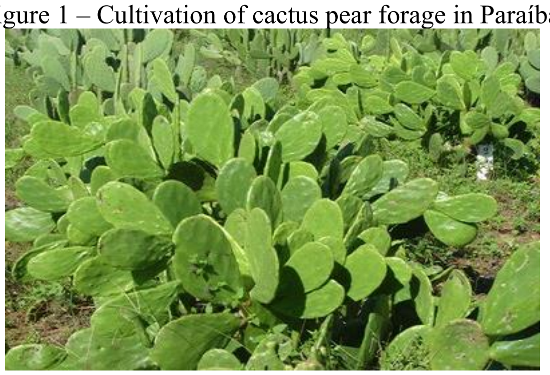
1.1.4 Brazil's Northeast Semi-Arid

Semi-arid region of Paraíba comprises an area of approximately 20,000 km² and is geoenvironmentally characterized by diversity of its landscapes and semi-arid condition that affects much of its territory and high spatial and temporal pluviometric variability inherent to this climatic type (SANTOS; SILVA; SRINIVASAN, 2007).

Due to irregular distribution of rainfall in the Northeastern semi-arid region, caatinga is an important forage resource. Thus, cultivation of perennial forage species, adapted to semi-arid conditions, is an important alternative for sustainability of production in this region. Cactus pear forage (*Opuntia fícus*) is metabolic CAM (Crassulacean Acid Metabolism) and presents high efficiency in the use of water (SANTOS *et a*l., 2011).

Cactus pear forage (*Opuntia ficus*), shown in Figure 1, is a culture originating in Mexico, and is currently cultivated worldwide (SANTOS *et al.*, 2011). Whereas *Opuntia cacti* originate from Mexico, they are cultivated in both hemispheres and on all continents except Antarctica (INGLESE; BASILE; SCHIRRA, 2002).

Figure 1 – Cultivation of cactus pear forage in Paraíba.



Source: www.liberdade96fm.com.br, 2014.

Cactaceae possess Crassulacean Acid Metabolism, which can be four to five fold more efficient in converting water to dry matter than most efficient grasses (STINTZING; SCHIEBER; CARLE, 2001).

Great diversity of uses and applications of cactus pear forage reveals versatility of this plant species, which despite being cultivated in semiarid for animal feed, does not have its full potential exploited. Consequently, excellent opportunities to improve social and economic indexes of this geographic space are being wasted, through generation of jobs, income, food supply and environmental preservation. Worldwide, forage palm is used in food as a source of energy (GALDINO *et al.*, 2010).

In addition to aforementioned characteristics, the fact that palm is a plant easily found in semi-arid regions makes it a kind of interest in research in which is needed of an easily obtainable biomass and persistent even in times of great drought.

1.1.5 Langmuir model

In 1918, Langmuir explained as a theory that adsorption occurs on a uniform, simple, infinite and non-porous surface (PORPINO, 2009). That model is based on hypothesis of movement of molecule adsorbed by adsorbent surface. The more molecules being adsorbed, the more uniform their distribution, which will form a monolayer that will cover entire surface (SENA, 2015).

Langmuir proposed model based on three hypotheses: I) adsorption can not go beyond coating with a layer; II) adsorption sites must be equivalent to each other and surfaces must be homogeneous; and III) ability of a molecule to be adsorbed at a given site is independent of occupation on neighboring sites (COELHO *et al.*, 2014).

With this, it was developed an equation (1):

$$\frac{q}{q_s} = \frac{bC}{1+bC} \tag{1}$$

On what: $\frac{q}{q_s}$ Is rate of adsorption; q_s is maximum adsorption capacity; b is Langmuir equation parameter and c is concentration of adsorbate in liquid phase.

It is considered as most efficient model in terms of isothermal representation where there is strong interaction between surface of adsorbent and solute for a single component. This model considers that there are a fixed number of sites in solid, these being equally energetic; therefore, it have same enthalpy of adsorption. Each site retains only one molecule of adsorbate (monolayer). Molecules adsorbed at nearby sites do not interact with each other and, at equilibrium, adsorption rate is equal to desorption rate (LIMA, 2010).

2 MATERIALS AND METHODS

Methodology used followed procedure performed by LIMA *et al.* (2014), which used mandacaru (*Cereus jamacaruna*) in particulate form to remove gasoline/diesel mixture, using adsorption process.

Ten rackets of cactus pear forage were collected. After collection, rackets were peeled and cut on cubes form, as shown in Figure 2.

Figure 2 – Cactus pear forage biomass cut on cubes form.

Source: Author collection (2014).

Material was left exposed in open air to be dried, remaining for three weeks. After this period, material was presented according to Figure 3.

Figure 3 – Cactus pear forage after drying.

Source: Author collection (2014).

Then, preparation of biomass on particulate form was started, in which process of comminution was carried out to powder form from cactus pear forage obtained in natura, using a blender. Material was classified in order to present particles with diameters ranging from 1 to 2 mm (Figure 4).

Figure 4 – Cactus pear forage biomass on powder form (diameter 1-2 mm).

Source: Author collection (2014).

Experiments were started to obtain kinetic curves, using 12 erlenmeyer flasks with 100 mL capacity for each battery of experiments (with contaminants lubricating oil and diesel oil). Procedure used was same for both water/lubricatingt oil and water/diesel oil mixtures, as described below.

It were added 40 mL of water and 12 mL of contaminant into erlenmeyer flasks. Mixture was shaken at 130 rpm on a shaker table, and 1.2 g of particulate biomass was added, sieved and dried at 5 minute intervals, with a time variation of 5 to 60 minutes, according to Figure 5.

Figure 5 – Shaking of mixture on shake table, under 130 rpm.

Source: Author collection (2014).

After each stirring time, flasks were subjected to filtration, with particulate biomass retained in the sieve and liquid phase drained into the beaker. Soon after, total water adsorbed in each sample was measured. At the end, adsorbent mass was determinated.

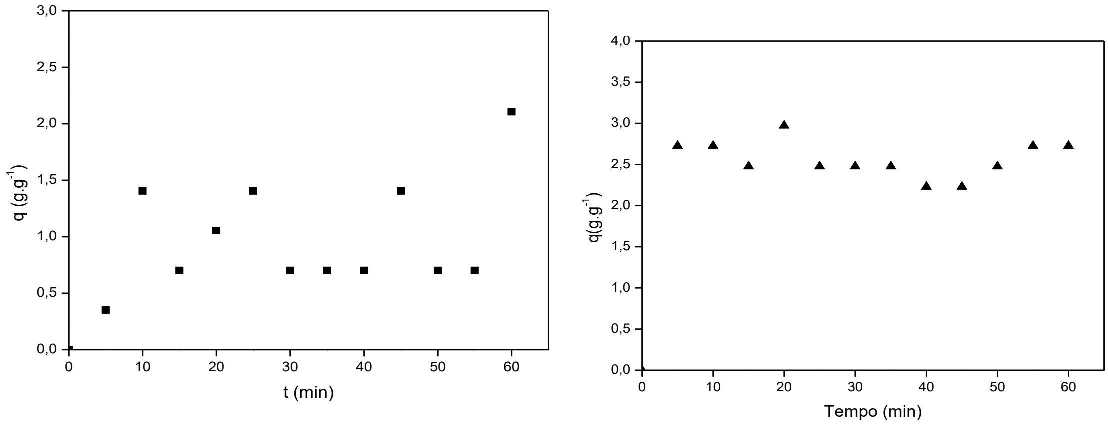
In study of equilibrium and adsorption isotherms, 12 erlenmeyer flasks with 100mL capacity were used, containing water contaminated with hydrocarbon, varying concentration of 5 to 60% contaminant, adding to the mixture 1.2g of particulate biomass, and maintaining them at 130 rpm on a shaker table for 60 minutes at room temperature. Samples was filtered, measuring contaminated mixture and biomass.

3 RESULTS AND DISCUSSION

3.1 ADSORPTION KINETICS

From collected data on proposed experiments, it was possible to construct curves referring to amounts of oil adsorbed by biomass in relation to contact time that was in agitation (Figure 6).

Figure 6 – Kinetic curves of adsorption of lubricating oil (a) and diesel oil (b).



(a) (b)

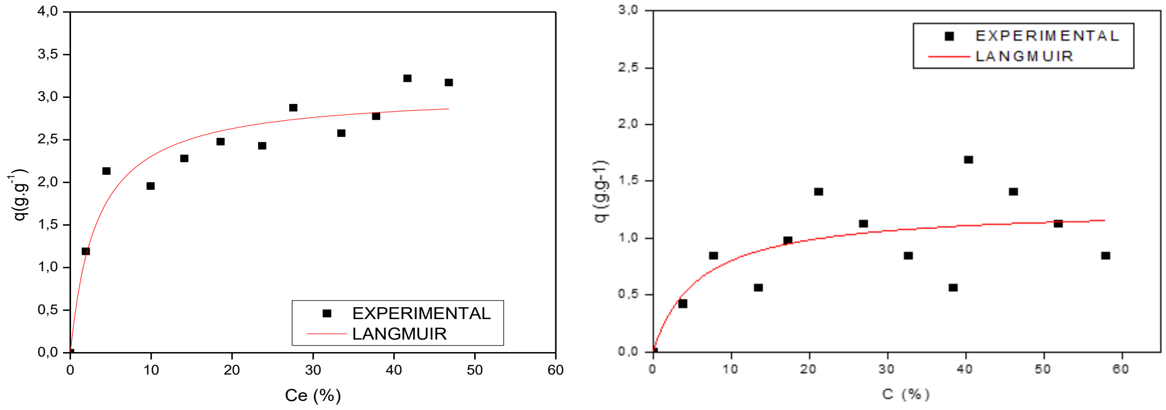
Source: Author collection (2014).

According to Figure 6, it can be said that adsorption kinetics to water/oil lubricating mixture was rapid, although adsorbed amount had not been elevated. At contact times of 15 and 40 minutes, highest contaminant adsorption values were reached (about 50%). In relation to water/diesel mixture, kinetics also showed to be rapid, with adsorption of contaminant starting at about 05 minutes after start contact. It is also observed that, in general, amount of adsorbate retained was low in times greater than 30 minutes, except for 60 minutes. Such behavior can be explained by molecular structure of diesel oil, constituted by large chains of hydrocarbons (15 to 18 atoms), that make it difficult to adsorb by biomass. There may have been breakage of hydrocarbon chains, forming chains of different sizes, which made adsorption non uniform after 30 minutes. This behavior was observed by Souza; Lima; Silva (2011), who used sugarcane bagasse as adsorbent biomass.

3.2 ADSORPTION EQUILIBRIUM

From collected data on experiments, it was possible to construct curve referring to amounts of oil adsorbed by biomass in relation to contaminant concentration in contact, applying Langmuir model (Figure 7).

Figure 7 – Adsorption equilibrium curve of lubricating oil (a) diesel oil (b) with cactus pear forage biomass applying Langmuir model.



(a) (b)

Source: Author collection (2014).

The equilibrium isotherm for water/lubricating oil mixture, presented in Figure XXXa, shows that, for lowest contaminant concentration, model allows a good adjustment, but for other concentrations same behavior is not observed. Adsorbed amounts in $g.g^{-1}$ were generally low and values close together.

Based on analysis of isotherms, it is possible to infer that cactus pear forage biomass presents efficiency to removal of contaminated lubricating oil at low concentrations, an efficiency observed by Albuquerque *et al.* (2014) for in natura vermiculite used to adsorption of lubricating oil in commercial form. However, as reported by Myamura (2011), after use, lubricating oil becomes contaminated and may undergo chemical reactions that result in a new compound and, in this sense, other studies that have studied removal of lubricating oil contaminated, using cactus pear forage as biomass do not found.

Regarding the biomass balance with water/diesel oil mixture (Figure 7b), it can be observed that it was reached, but with low amounts of adsorbed contaminant. At concentrations of 20 and 40%, biomass had higher adsorption efficiency. In general, the behavior of equilibrium curve oscillated between high and low adsorption values. Such behavior can be attributed, according to Brandão *et al.* (2006), at the end of monolayer, with adsorption occurring in multilayers, but on a lesser extent.

Non homogeneity of behavior observed by studied system may have been influenced by molecular structure of diesel oil. According to Aguiar (2013), chemical composition of diesel is characterized by light fractions and heavy fractions of hydrocarbons, with insertion of most different additives. Such composition, with chains of different sizes, can favor oscillation in adsorption by means of cactus pear forage biomass.

4 CONCLUSIONS

From analysis of kinetic curves of adsorption and equilibrium study it is possible to state that kinetics of adsorption was fast for both mixtures. The highest amount of adsorption of contaminant to water/lubricating oil mixture was observed over course of 20 min (approximately 3 $g.g^{-1}$). In spite of low values of adsorption confirmed by kinetic and equilibrium graphs in adsorption for water/diesel mixture, 50% of adsorbed contaminants were obtained in some points, which can characterize forage palm as a relatively efficient adsorbent.

It was observed that internal structure of contaminant exerts great influence on its adsorption by cactus pear forage biomass. The more complex chains of diesel oil decreased adsorption efficiency when compared to molecular structure of lubricaing oil, which presented higher values of adsorption by cactus pear forage.

The study of cactus pear forage as biomass for adsorption of organic contaminants is still in its beginning. Although results do not point to a very high adsorption of contaminants, this biomass can be characterized as an efficient, economical and sustainable alternative to removal of effluents with low concentrations of contaminated lubricating oil.

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