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Parametric Study and Empirical Modeling for the Equilibrium and Kinetic Adsorption of Milk Organics onto Stone Cutting Particles

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Abstract

The present paper investigates the treatment of dairy wastewater by adsorption on stone cutting solid waste. It implements the innovative concept of "treating waste by waste." Batch adsorption experiments were performed for obtaining kinetic curves of percentage reduction in COD. The treatment efficiency is investigated as functions of the operating parameters including: stirring rate, pH, particle dosage and contact time.

Both the rate of adsorption and the equilibrium removal efficiency increase with increasing particle dosage. Increasing the stirring rate increases the adsorption rate, while it has no effect on equilibrium efficiency. The effect of pH is found to be unique; the adsorption occurs mainly at a pH value of around 6. Pseudo second order model is found to fit experimental data.

Key words: Adsorption, COD, Equilibrium, Kinetics, Dairy, Stone, Wastewater.

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1. Introduction

Dairy industry and stone cutting are two main industries in Palestine. Dairy industry is a major food processing industry which needs water in all manufacturing processes. Dairy industry generates wastewater effluents characterized by high Biological Oxygen Demand (BOD) and Chemical Oxygen Demand (COD). When released into valleys and open areas, major pollution problems are faced; it percolates through soil and thus polluting the ground water. Also, stone cutting industry generates large amounts of wastewater, which contains suspended particulate matter that affects soil and ground water.

In Palestine, dairy industry contributes by 6.8% in the food industry sector (Economy, 2004). On the other hand, Palestinian stone and marble industry constituted approximately 4% of world production. It contributes approximately 5.5% to gross domestic product and employs 15,000 workers with annual sales reaching 270 million dollars according to 2006 industrial survey (Economy, 2006).

Dairy wastewater contains massive amounts of organic matter (proteins, lactose and fat), sewage fungus, and inorganic compounds (Nitrogen, Phosphorus and Ammonia). The average values of BOD and COD in the dairy effluents were reported to be 1941 ± 864 ppm, 3383 ± 1345 ppm, respectively (Shete and Shinkar, 2013). The pH was reported to be 7.9 ± 1.2 and TSS was reported to be 831 ± 392. There have been no noticeable efforts for controlling dairy industry wastewater in Palestine. Currently, most of the industrial wastewater in Palestine is discharged directly into sewer system (62.8%). The rest (37.2 %) is discharged through cesspits

For stone industry, it has numerous major environmental impacts. Al-Jabari has previously emphasized its environmental impacts on agriculture, ground water and human beings(Al-Jabari, 2002). It is estimated that the stone cutting industry in Palestine annually uses about 0.5 million cubic meters of water. Thus, the resulting wastewater contains suspended solid particles (slurry). Then, these particles are separated from wastewater using various techniques (Al-Jabari, 2002; Al-Jabari and Sawalha, 2002) and the resultant separated particles are transported to open areas (Al-Jabari et al., 2012). It is estimated that this industry annually generates approximately 0.7-1.0 million tons of this slurry waste.

Various local trails tried to control or reduce the impact of stone industry. For example, sludge recycling projects. Where, the slurry powder can be used in PVC pipes, tiles and artificial stone production (Al-Joulani, 2006; Al-Joulani, 2007). In addition, Al-Jabari used stone cutting wastewater as a treatment option for local leather tanning industry (Al-Jabari, 2002).

Worldwide, there are various treatment methods for wastewater effluents from dairy industry. These include; activated sludge, trickling filters, sequence batch reactors, anaerobic sludge blanket, nanofiltration and others. These techniques are complicated, expensive, energy consuming and unable to reach effluent discharge standards of 50 ppm BOD and 250 ppm COD according to World Bank restrictions (Shete and Shinkar, 2013).

One of treatment methods is adsorption. Organic material in dairy wastewater can be adsorbed onto various solid adsorbents. Previous studies have confirmed the technical feasibility of adsorbing organics on various adsorbents. These

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adsorbents include; low molecular weight crab shell chitosan (Devi, 2012), Activated Carbon Commercial grade (ACC) (Kushwaha et al., 2010), Bagasse Fly Ash (BFA) (Kushwaha et al., 2010), acid mine drainage sludge (Wang et al., 2013), clay particles (Al-Jabari, 2016), marlstone (Al-Jabari, 2016) and Neem leaves powder (Sivakumar and Christyno, 2012). Investigated parameters in these studies included: pH, particle dosage, contact time, stirring rate and initial concentration of organics.

Various empirical models were used for adsorption kinetics. These included Elovich and Lagergren pseudo-first order and second-order models order, and second-order models. Such models were reviewed by Ho (Ho and McKay, 1998). Adsorption kinetics based on an nth-order kinetic model, and a double exponential model were used by Tosun (Tosun, 2012). Second-order adsorption models were reviewed by Ho [9 From marlstone]. First order model was used by Elagroudy to estimate the mass adsorbed in ith minutes (Mi) [10 From marlstone]. A full review of modeling papers is available in the literature [11 From marlstone]. Two new theoretical models for adsorption kinetics have been published recently. These include a mass transfer model [maher mass] and a model based on Langmuir kinetics [maher langmuir].

There are urgent needs to reduce environmental impacts associated with dairy wastewater. Since there are no noticeable efforts to control dairy wastewater, adsorption is believed to be the simplest solution for reducing COD in dairy industry wastewater. It is most preferred when a low cost abundant adsorbent is used. A recent paper by the present authors had demonstrated the technical feasibility of treating dairy wastewater with various local abundant adsorbents (Al-Jabari et al., 2015). These local natural adsorbents were used also to treat other types of industrial wastewaters, such as leather tanning wastewater (Al-Jabari et al., 2012, Al-Jabari et al., 2009a; Al-Jabari et al., 2009b).

The aim of the present experimental study is to investigate the effects of various operating parameters on adsorption process for treating wastewater from diary industry with solid waste from stone cutting industry. These parameters include solid content, pH, contact time, stirring rate and organics initial concentration.

2. Materials and Methods

2.1 Materials

Samples of stone cutting solid waste are obtained from a local factory in Hebron, that does not involve flocculation-sedimentation process for its wastewater treatment (i.e., no use of polymeric flocculating agents) see (Al-Jabari, 2002; Al-Jabari and Sawalha, 2002). Flocculated particles are not inefficient for adsorption (Al-Jabari et al., 2015). The obtained solid samples are dried in an oven at 120oC. The size of solid waste particles is determined to 34 µm,using settling test method. The obtained particle size agrees with results obtained by Al-Jabari (2002). The used solid particles contain 85% of calcium carbonate (obtained by reverse titration).

Real samples of dairy wastewater are obtained from a local dairy factory (AL-Jebreni Company, Hebron, Palestine). The measured COD of the wastewater sample was 5200 mg/L, with a percentage difference of 3%. No physical pretreatment of the wastewater was made. Wastewater samples are stored in a refrigerator at 4 oC. An amount of 2 mL of concentrated sulfuric acid (18M) is added to each one liter of wastewater to prevent natural biodegradation, according to the standard requirements(Clesceri et al., 1998). It is diluted at a ratio of (1:10) by adding distilled water. The initial COD and BOD of the wastewater used in the adsorption process are about 500 mg/L and 300 mg/L, respectively.

Chemical reagents used include Potassium Hydrogen Phthalate, Potassium Dichromate, Sulfuric Acid 99% purity, 1.1 Phanthroline and Ferrous Sulfate. All chemicals are from Sigma Aldrich, through Alfa Aesar Company in Palestine.

2.2. Methodology

A volume of 100 mL of wastewater is mixed with a required mass of stone cutting particles, for batch adsorption experiments. Batch adsorption experiments at ambient room temperature (22oC) are carried out in stirred vessels. At certain time intervals, small samples of wastewater are then taken from the adsorption vessel and analyzed using standard COD test procedure (Clesceri et al., 1998). Determination of measured COD and estimated BOD is illustrated in a previous paper (Al-Jabari et al., 2015).

2.3. Data Analysis

The efficiency of the adsorption process is obtained from the percentage COD reduction (or removal efficiency), as given by the following equation:

$$\text{Percentage removal efficiency} = \frac{\text{COD}_o - \text{COD}_t}{\text{COD}_o} \times 100\% \qquad (1)$$

where CODo is the initial COD of wastewater (mg/L), CODt is the obtained COD after adsorption at certain time (mg/L).

The surface concentration of organics on stone particles (qtin mg/g) is obtained from batch massbalance for adsorption process as follows:

$$q_t = \frac{V(\text{COD}_o - \text{COD}_t)}{m} \qquad \text{........................(2)}$$

where m is the mass of adsorbent (mg) and V is the volume of wastewater used in the adsorption experiment (100 mL).

3. Results and Discussion

The main results of the present paper are presented here as curves of COD, qt and percentage reduction in COD as functions of time. The validity of monitoring COD reduction in dairy wastewater for evaluating performance of wastewater treatment processes has been demonstrated experimental in previous works (Shete and Shinkar, 2013). The technical feasibility of organics removal from dairy wastewater by its treatment using stone cutting solid waste particles was confirmed previously by the authors of the present paper (Al-Jabari et al., 2015). The effects of various parameters on adsorption process are presented; these included contact time, bulk motion, solid content, pH and organics concentration.

3.1. Adsorption Kinetics

Typical adsorption kinetics is presented as a plot of qt versus time, as presented in Figure 1. It shows two groups of experimental data obtained from two identical adsorption experiments (circles and triangles), with a particle dosage of 5 g/100 mL, and at a temperature of 22°C, pH= 6 and a stirring rate of 250 rpm. It clearly confirms the reproducibility of data and the validity of the used experimental procedures.

Figure 1 indicates that the organic load on the surface of stone cutting particles increases with time, as a result of the adsorption of organics from the dairy wastewater. At equilibrium, the rate of adsorption equals the rate of desorption, and thus no further net change in qt occurs, resulting in constant equilibrium value. Figure 1 indicates that the adsorption process is relatively fast. Equilibrium is approached within 3 hours. A similar kinetic behavior was obtained on the same type of adsorbents, in a previous study on treating leather tanning wastewater with stone cutting solid waste (Al-Jabari et al., 2012). However, organic adsorption seems to be faster than chromium adsorption.

The obtained experimental kinetic adsorption curve is modeled using pseudo first order and second order rate equations, given in Eqn.4, Eqn.5 and Eqn.6, presented in linear forms:

$$\log(q_e - q_t) = \log q_e - \frac{K_1}{2.303} t \quad \text{.......................(3)}$$

$$\frac{t}{q_t} = \frac{1}{V_o} + \frac{1}{q_e} t \quad \text{.......................................(4)}$$

$$V_o = K_2 q_e^{2} \quad \text{...(5)}$$

where K1, K2 in (hour-1) are the pseudo first order and second order rate constant for the kinetic model, respectively, qe and qtin(mg/g) are the adsorption capacities at equilibrium and at a time tin(hours), respectively, and Vo is the initial adsorption rate.

The solid curve in Figure 1 is obtained from the pseudo second order model, while the dashed curve is from the pseudo first order model. Obviously, the second order model provides a better fit for the experimental results. This is in agreement with the findings in previous studies: pseudo second order model provided excellent fit for the adsorption kinetics of organic pollutants. These cases included the adsorption kinetics for the adsorption of methylene blue on high lime fly ash (Keleşoğlu et al., 2012), the adsorption kinetics of dairy proteins on silica (Sarvi et al., 2014), the adsorption kinetics of dairy organics on rice husk (Pathak et al., 2016), and the adsorption kinetics of organic pollutants in dairy wastewater on soil particles (Al-Jabari, 2016).

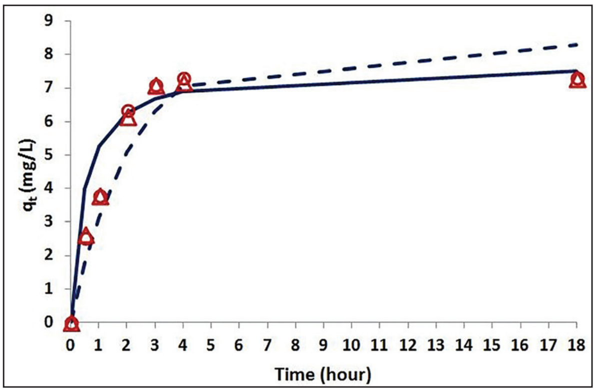

Figure 1. Experimental kinetic curve of qt (organics load on the adsorbent) and model curves using pseudo first order rate equation (dashed line) and second order (solid line), for adsorption experiments using stone cutting solid waste with a particle dosage of 5 g/100 mL at a temperature of 22°C, pH= 6 and stirring rate= 250 rpm.

3.2. Effect of Stone Particle Dosage

Increasing the particle dosage has a major effect on the final removal efficiency, since it increases the total available surface area for adsorption. Figure 2A shows the adsorption kinetic curves for different cases of stone particle dosages indicated in the figure caption. All experiments are performed at pH=6, a temperature of 22 oC and a stirring rate of 250 rpm. Table 1 summarizes the final-equilibrium removal efficiency for various particle dosages. With high particle dosage of 10 g/100 mL, the obtained percentage COD removal (83%) is relatively high, and the treated wastewater complies with the World Bank standards.

Figure 2A also shows that changing particle dosage affects the adsorption rate: Increasing the particle dosage increases adsorption rate; it decreases the time needed to approach equilibrium, and increases the slope of the kinetic curve at each time. This is attributed to the fact that with more particles in liquid, more collisions with particle surfaces occur and thus faster adsorption is obtained. Increasing solids to liquid ratio increases the mass transfer coefficient and decreases the characteristic time needed to approach equilibrium.

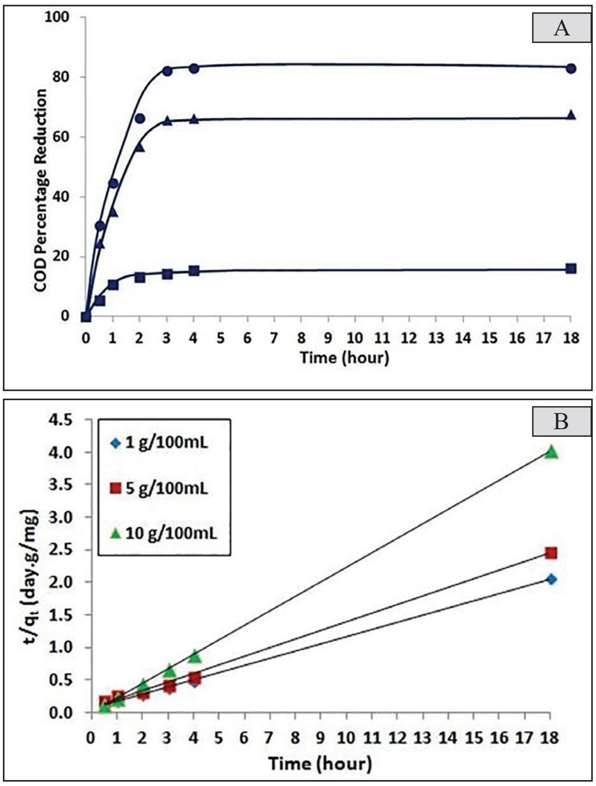

Figure 2. (A) Percentage COD removal as a function of time for different particle dosages (squares for 1 g/100 mL, triangles for 5 g/100 mL and circles for 10 g/100 mL), all at pH=6, 22 °C and a stirring rate= 250 rpm. **(B)** Second order kinetic model for the adsorption of organic molecules onto stone cutting particles for different particle dosages.

The obtained experimental kinetic adsorption curves are modeled using pseudo second order rate equation, given in Eqn.5 and Eqn.6, declared in linear forms and presented in Figure 2B.With the second order model, the plot of t/qt versus time is linear, with a positive slope of 1/qe. Obviously, the model provides good fit to the experimental data. Obtained model and fitting parameters are listed in Table 1.

Table 1. Obtained model and fitting parameters with pseudo second order model for the adsorption of organic molecules onto stone cutting particles and experimental equilibrium removal efficiency for various particle dosage, at pH=6, a temperature of 22°C and stirring rate of 250 rpm.

Dose (g/100mL)	Experimental	Pseudo Second Order Parameters				
	Percentage Reduction	qe (mg/g)	Vo	K2 (hour-1)	R2	SD
1	18%	9	19.53	0.241	0.9978	0.17
5	68%	7.51	16.23	0.288	0.9964	0.15
10	83%	4.48	3.3*1015	1.6*1014	1	0

3.3. Effect of Bulk Motion

The effect of increasing stirring rate is presented in Figure 3 (for similar conditions as in above cases in Figures 1 and 2. At low stirring speed of 70 rpm, equilibrium is approached within about 4 hours, which is larger than the time period for the case with 250 rpm (about 3 hours). It is believed that the liquid side mass transfer resistance controls the process. Increasing bulk motion increases the volumetric convective mass transfer coefficient and thus reduces the time needed to reach equilibrium. Thus, the adsorption rate increases with bulk motion. This is in agreement with previous work (Al-Jabari, 2016; Al-Jabari, 2016).

The final removal efficiency does not change with bulk motion, since it is characterized by the equilibrium adsorption capacity. Adsorption capacity is a surface property and does not depend on the surrounding hydrodynamic conditions. It is obtained from equilibrium isotherm.

These results are in agreement with various previous studies, where increasing stirring rate increased adsorption rate and had no effect on the final adsorption capacity (Asgari et al., 2013, Mehmet Doğan and Özmetin, 2006).

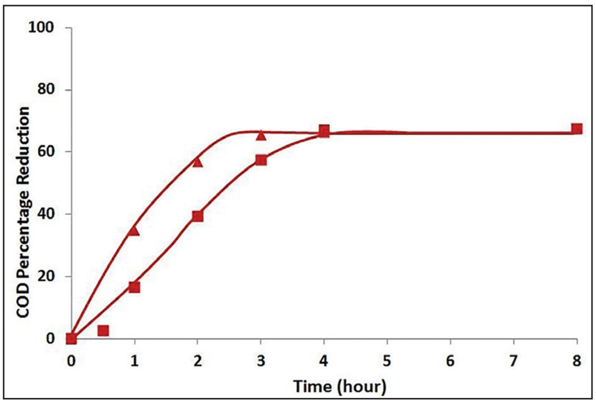

Figure 3. Percentage COD reduction as a function of time, at two stirring rates (squares for 70 rpm and triangles for 250 rpm) at pH=6, at a temperature of 22°C and a particle dosage of 5g/100 mL

The above fast adsorption rate was obtained when the system was kept under stirring (suspended particles in the vessel). When adsorption vessel is left unstirred (particles are settled at the bottom of the vessel), the adsorption process is extremely slow. Figure 4A shows results obtained for adsorption experiments at the same conditions as that in Figure 1 but with no stirring. Also, Figure 4A compares COD Kinetic curves for adsorption on soil and marlstone particles (Al-Jabari, 2016; Al-Jabari, 2016). With all types of adsorbents, nearly, 9 days are required to reach equilibrium. This emphasizes the hypothesis that the adsorption of organics on these mineral particles is mass transferred controlled. When the system is not stirred, the mass transfer occurs by diffusion which is a slow process compared with convection mass transfer (when the system is stirred).

With stone cutting particles, the COD value with stagnant particles has nearly the same value as that for the case of stirring. This is attributed to fact that the adsorption capacity is a surface property obtained from equilibrium isotherm, and does not depend on the hydrodynamic conditions.

Both soil and marlstone have similar adsorption behavior, but stone cutting particles have higher adsorption rate and removal efficiency. For the purpose of comparison, results from similar adsorption experiments, utilizing red clay soil (90.5 µm) and marlstone particles (53 µm), are summarized in Table 2. It lists the maximum adsorption rate and COD percentage reduction, for each adsorbent. Stone cutting particles are able to reduce COD twice more than marlstone. Soil particles exhibit one fifth of the maximum adsorption rate of stone cutting particles.

Table 2. Comparison between natural mineraladsorbents and their COD removal efficiencies, maximum adsorption rate and obtained model and fitting parameters with pseudo second order model for the adsorption of organic molecules onto natural adsorbents particlesperformed at stagnant conditions, pH=6, 22oC and using a dose of 5 g/100mL.

Adsorbent	Experimental	Pseudo Second Order Parameters				
	Maximum Specific Rate (mg/L. day)	Percentage Reduction	qe (mg/g)	Vo	K2 (day-1)	R
Soil	-20.72	27%	3.74	1.12	0.081	0.9874
Marlstone	-46.21	32%	4.12	1.31	0.077	0.9927
Stone cutting solid waste	-97.21	66%	8.69	2.87	0.038	0.9944

For the three kinetic curves (for soil, marlstone and stone cutting solid waste) in Figure 4A, the plots of t/qt versus time are linear, with positive slopes of 1/qe. The resulting lines for the second order model are plotted in Figure 4B. The obtained model and fitting parameters are listed in Table 2. Obviously, the pseudo second order model gives relatively good fitting to the experimental data. As listed in Table 2, both marl and soil adsorbents have higher pseudo second order rate compared to stone cutting solid waste. On the other hand, stone particles have a higher adsorption capacity (higher qe)

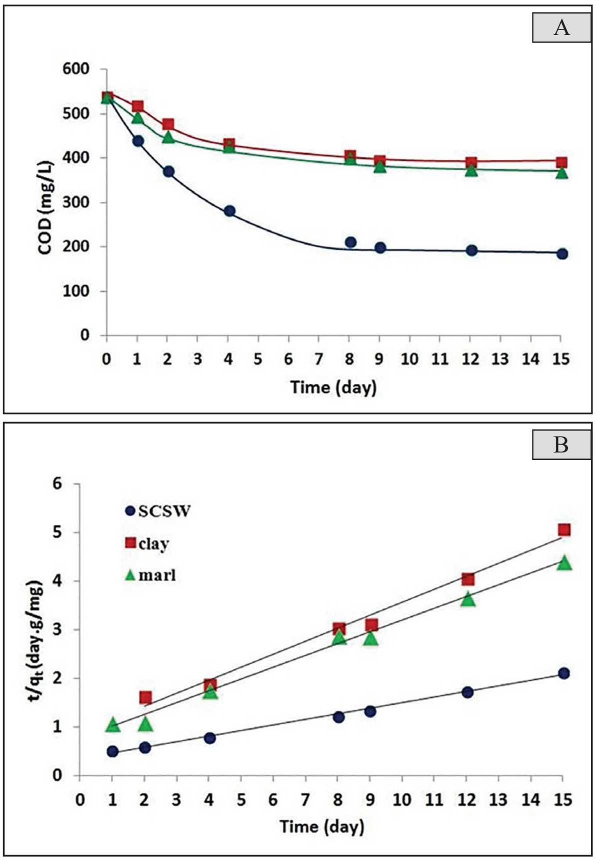

Figure 4. (A) Adsorption kinetics (COD versus time) for static conditions (no stirring) with a particle dosage of 5 g/100 mL of adsorbent, at a temperature of 22ºC and pH= 6 (squares for soil, triangles for marlstone and circles for stone cutting). **(B)** Second order kinetic model for the adsorption of organic molecules onto various mineral adsorbents for stagnant particles

3.4. Effect of pH

It is well known that adsorption on surfaces is pH dependent. Figure 5 shows the obtained results of equilibrium removal efficiency (circles for stone particles) as a function of solution pH using a particle dosage of 5 g/100 mL, at a temperature of 22ºC and a stirring rate of 70 rpm. A wastewater pH range between 2 to 12 is obtained by adjusting pH using concentrated hydrochloric acid and sodium hydroxide. Samples using the same mass (5 g/100 mL) with different pH values were stirred for 18 hours (sufficient time to reach equilibrium) and allowed to settle for 24 hours. Then the final equilibrium value of COD is measured. Obviously, for stone cutting particles, low COD percentage reduction is obtained at extremely acidic and alkaline conditions. There is a limited pH range for adsorption, which is nearly 5-7, with a maximum removal efficiency of 68% at pH=6 (for particle dosage of 5 g/100 mL).

This obtained type of pH dependence of organics uptake efficiency by stone cutting particles is unique .For adsorption on charged particles (like stone particles of the present work); usually the removal efficiency will be high at alkaline conditions and small at acidic conditions. In this case, the adsorption process is efficient mainly at pH value of around 6.

It is believed that this unique behavior of stone cutting particles is associated with physical adsorption, in which Van der Waals forces bond solute to the surface. This occurs when solution pH eliminates repulsion forces associated with high pH values. However, in the previous work of adsorption of positively charged trivalent chromium ions(Al-Jabari et al., 2009a), the surface charge of stone particles is essential in the adsorption mechanism, and thus high pH values resulted in attractive forces and yielded high adsorption efficiency. At low pH values, (obtained by the addition of sulfuric acid), a chemical conversion of stone particles occurs, i.e., reaction of H_2SO_4 with $CaCO^3$ producing calcium sulfate and releasing CO_2. The resulting product (yellow colored) does not have affinity for adsorption as stone particles, and thus zero removal efficiency is observed.

Also, Figure 5 compares the pH dependence for other mineral adsorbents: soil (squares) and marlstone (triangles) (Al-Jabari, 2016; Al-Jabari, 2016). Soil particles seem to favor acidic conditions to adsorb organic molecules. On the other hand, adsorption of organic matter on marlstone particles occurs in neutral to alkaline conditions. Soil in its nature is alkaline, and from Figure 5, soil pollution with organics is of small probability. However, if soil is acidic or contains a fraction of marlstone, it will be more suspected to pollution from dairy wastewater.

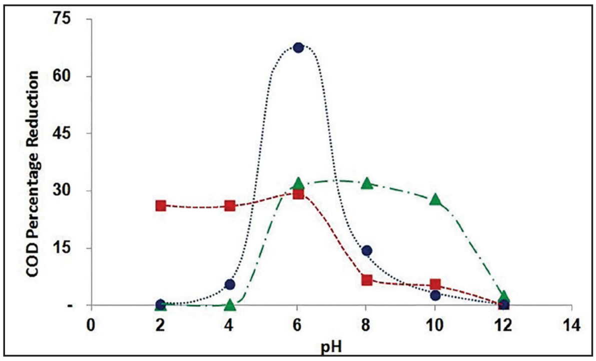

Figure 5. Equilibrium removal efficiency as a function of solution pH using a particle dosage of 5 g/100 mL, at a temperature of 22ºC and a stirring rate of 70 rpm (squares for soil, triangles for marlstone, and circles for stone cutting).

3.5. Adsorption Isotherm

Figure 6 presents the adsorption isotherm as a plot of equilibrium concentration in the solution versus surface concentration on the stone particles, for equilibrium experiments with a particle dosage of 10 g/100 mL, pH= 6, and at a temperature of 22ºC. Obviously, the isotherm is linear. This linearity supports the research hypothesis that physical adsorption occurs with a mass transfer process.

These results indicate that a kinetic adsorption model based on mass transfer rate equation can be developed in a similar fashion as the mass transfer desorption model developed by Al-Jabari and Weber for solute desorption from solid surface into fluid (Al-Jabari, 1999). In such a case, the only difference is in the initial conditions, i.e., at zero time: the surface concentration (qt) is zero and the dimensionless bulk fluid concentration (COD/CODo) is 1.

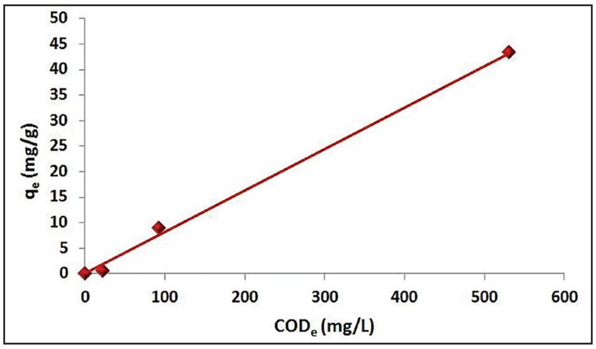

Figure 6. Adsorption equilibrium isotherm as a plot of equilibrium concentration in the solution versus surface concentration, for equilibrium experiments with a particle dosage of 10 g/100 mL, pH= 6, and at a temperature of 22°C.

Conclusion

The present paper demonstrates that the solid waste particles from stone cutting industry can reduce the organic load (COD) in the wastewater from dairy industry, in an efficient adsorption process. The process is mass transfer controlled with linear equilibrium isotherm. The rate of adsorption increases with increasing bulk motion. When the system is not stirred an extremely slow adsorption process occurs. Pseudo second order model is found to fit experimental data. Equilibrium removal efficiency is high at a pH of 6. It increases with increasing particle to wastewater ratio. Adsorption on stone cutting particles is more efficient than that on other similar mineral particles (marlstone and soil).

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