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STUDY OF MOXIFLOXACINADSORPTION ONSPENT COFFEE GROUNDS

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ABSTRACT

The adsorption of moxifloxacin (MOX) by the spent coffee grounds (SCGs) was investigated. The effect of solution pH and the amount of SCGs were also studied. The optimum solution pH and amount of SCGs were 6 and 40 mg, respectively. The adsorption isotherms were well described by the Henry model. This suggested that the strong interaction of MOX with the SCGs. Therefore, SCGs, as a green, environmental-friendly adsorbent, can be applied to the adsorption of contaminants in environment.

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INTRODUCTION

Fluoroquinolones, which act as inhibitors of DNA topoisomerase II, exhibit both strong bactericidal and sterilizing activities against the *Mycobacterium tuberculosis complex* Among these drugs, moxifloxacin (MOX)is one of the drugs with high efficacy against tuberculosis (Brahmadhi *et al.*, 2021). However, MOX is considered highly harmful to plants, algae, and bacteria, as well as hazardous to animals and human (Sija Arun *et al.*, 2020). Therefore, the monitoring of MOXin various environmental samples became crucial.In recent years, biomass is used as sorbents to adsorb drugs due to their big surface area, low-cost usage, feasible generation and excellent adsorption properties. Biomass contains product, byproducts, residues and waste fromagriculture, forestry and industrial processes are undertaking efforts for the utilization of sorbents for various contaminants (Elgharbawy *et al.*, 2020). Spent coffee grounds (SCGs) can also been used as an inexpensive adsorbent for removal of dyes, heavy metals, and pollutants (Anastopoulos *et al.*, 2017). In this study, spend coffee grounds was applied to adsorb MOX. The adsorptive kinetics and adsorptive isotherm of SCGs for MOX were investigated. The results were analyzed by high performance liquid chromatography (HPLC).

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EXPERIMENTAL

Chemicals and Materials

MOX was purchased from Sigma-Aldrich (Steinheim, Germany), high performance liquid chromatography-grade methanol (MeOH) and acetonitrile (ACN) were provided by J&K Chemical (Beijing, China). NaH_2PO_4, H_3PO_4, NaOH, and other affiliated chemicals were all obtained from Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China). All solvents and chemicals were of analytical grade and used without further purification unless otherwise specified. HPLC-grade water was obtained by purifying demineralized water in a Milli-Q system (Millipore, Bedford, MA, USA), and was used throughout the work.

Apparatus and software: For chromatographic separation, an Agilent 1260 HPLC system (Agilent Technologies, CA, USA), equipped with a quaternary pump, a degasser, a column compartment, and a UV detector were used. Separation was performed on a Pursuit 5 C18, 5 µm, 4.6 mm ×150 mm column. The injection volume was 20 µL and the ultraviolet (UV) detector was set at 270 nm. The mobile phase consisted of 0.2% acetic acid and ACN with a ratio of 85:15 (v:v)at a flow rate of 1.0 mL/min. All the samples were passed through microporous nylon filters of 0.45 µm pore sizes in diameter (Pall Corporation, USA). An Ion 510 pH meter (Ayer Rajah Crescent, Singapore) was used to monitor pH adjustment. A centrifuge (Xiangyi, Hunan, China) was used for sample preparation.

Preparation of standard: Standard stock solution containing 1000µg/mL of MOX was prepared by dissolving the required amounts of the standard in MeOH. It was stored in a refrigerator at 4 °C. Working solutions were prepared from the stock solutions by dilution with appropriate amounts of Milli-Q water.

Adsorptive performance experiment and isotherm modeling: Each desired MOX concentration used in batch experiments was prepared by appropriately diluting the stock solutions with 10 mmol/L NaH_2PO_4 and successive dilutions. SCGs were added into 5 mL MOX solution with a fixed concentration. All the adsorption experiments were performed in conical flasks under ultrasonic bath for 0.5 h to achieve an adsorption equilibrium. After adsorption, all solutions were filtered through 0.45 µm membrane filters and analyzed by HPLC. The adsorption capacity (q_e, mg/g) of SCGs for MOX was calculated by the following formula (Wang *et al.* 2017):

$$q_e = \frac{(C_0 - C_e) \times V}{m} \quad (1)$$

where C_o and C_e are the initial and equilibrium concentrations of each MOX (µg/mL), respectively; V is the volume of adsorption solution (5 mL); m is the weight of SCGs(mg). The impact of initial solution pH on MOX adsorption efficiency were conducted by adding 40 mg SCGsinto each MOX solution (80 µg/mL, 5 mL) with ultrasonic bath assisting for 0.5 h. The pH value was adjusted by NaOH or H_3PO_4 solution (0.1 M) ranged from 3.0 to 9.0. The impact of SCGs amount on the adsorption efficiency was tested by adding different amount of SCGs (5–100 mg) to each MOX solutions (pH=6, 80 µg/mL, 5 mL) with ultrasonic bath assisting for 0.5 h. The impact of initial concentration on the adsorption efficiency was tested by adding 40 mg SCGs to different concentrations of each MOX solutions (5–250 µg/mL, pH=6, 5 mL) with ultrasonic bath assisting for 0.5 h. In this work, three common isotherm models, such as Henry model (HM), Freundlich model (FM) and Langmuir model (LM), see Equation (4), (5) and (6), respectively(Jung *et al.* 2013; Chang *et al.* 2018), were applied to interpret the adsorption isotherm data.

$$q_e = K_D c_e \quad (4)$$

$$q_e = K_F C_e^{\frac{1}{n}} \quad (5)$$

$$1/q_e = 1/Q_m + 1/K_L Q_m c_e \quad (6)$$

K_D (L/g) is the Henry sorption coefficient, K_F(L/mg) is the Freundlich sorption coefficient, and n is the indicator of isotherm nonlinearity. Q_{max}(mg/g) is the Langmuir sorption capacity, and K_L (L/mg) is the sorption affinity parameter in the Langmuir model.

RESULTS AND DISCUSSION

Effect of pH: Solution pH is a major influencing factor in adsorption process, since it can alter the surface charge of adsorbents and the speciation distribution of the analytes in solution. Figure 1 illustrates the effect of initial pH on MOX uptake by SCGs with pH ranging from 3.0 to 9.0. It could be found that q_e of MOX increased as pH value increased from 3 to 6, decreased as pH value increased from 6 to 8, increased little as pH value increased from 8 to 9, and the q_e value was biggest when pH was 6.These phenomena may be resultant from the surface charge of SCGs and the speciation of MOX at different pH values. It is reported that MOX have three pK_a values in solution ($pK_{a,1}$, $pK_{a,2}$ and $pK_{a,3}$) because these compounds possess a piperazinyl substituent and a carboxylic group. As two protonation/deprotonation equilibria are involving in the piperazinyl substituent of these compounds, two pK_a values are expected to associate with the piperazinyl substituent (Lina *et al.* 2004). The $pK_{a,1}$, $pK_{a,2}$ and $pK_{a,3}$ for MOX was 5.05, 6.25 and 9.29, respectively(Langlois *et al.*, 2015; Doorslaer *et al.*, 2011). When pH was lower than 6, positive species were the main species of MOX in solution. If there were negative charges on the surface of SCGs, the adsorption was strong, the value of q_e would be big. The result showed in Figure1 indicated this phenomenon. When the pH was bigger than 6, negative species were the main species of MOX in solution. The adsorption would be weak, the value of q_e would be small. This was also indicated in Figure 1. Therefore, pH 6 was selected as the best solution pH.

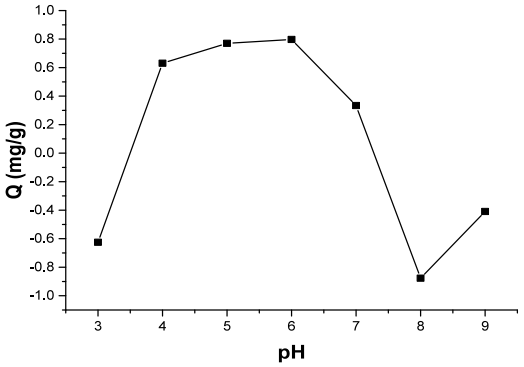

Figure 1. Effect of solution pH on adsorption capacity

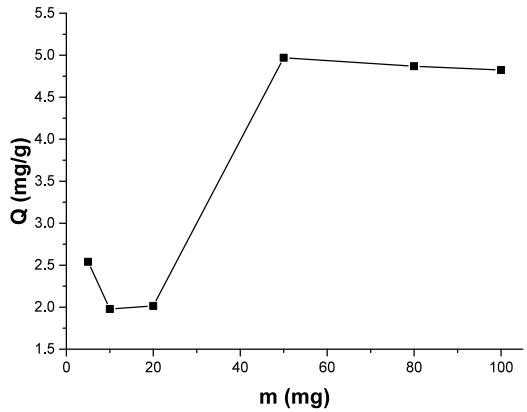

Figure 2. Effect of SCGs on adsorption capacity

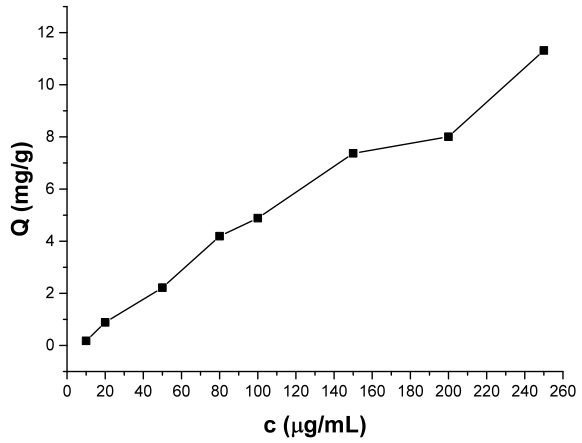

Figure 3 Effect of MOX concentration on adsorption capacity

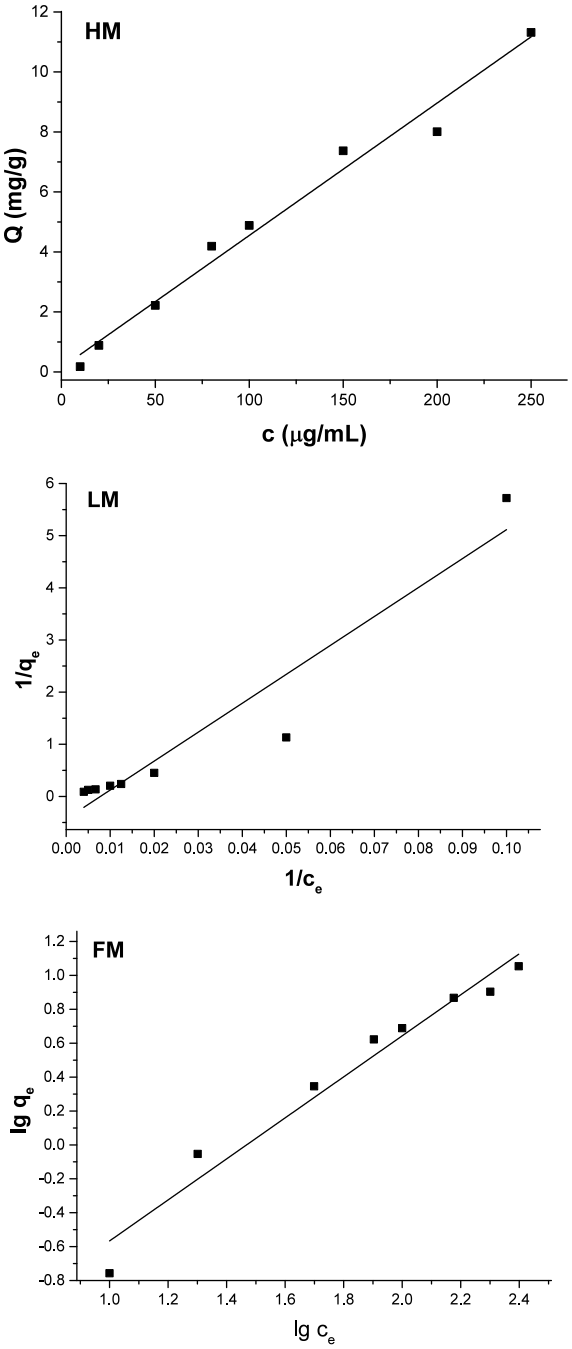

Figure 4. Fitted curve of HM, LM and FM

Table 1. Adsorption parameters of HM, FM and LM for MOX onto SCGs

HM			FM			LM	
R^2	K_D	intercept	R^2	K_F(L/kg)	1/n	R^2	K_L
0.9785	0.00247	0.13635	0.9587	0.01673	1.2100	0.9065	-0.4311

Effect of SCGs usage amount: The amount of SCGs in the solution was varied from 5 to 100 mg, see Figure 2. The results revealed the q_e value increased rapidly at first, then reached equilibrium at 40 mg. Thus, 40 mg was selected.

Adsorption isotherms: The adsorption isotherm is crucial in understanding the adsorption capacity of SCGs and very useful to describe how the MOX distribute on the SCGs when the adsorption process reaches an equilibrium state. Figure3 showed uptake of MOX by SCGs. As can be seen, q_e of SCGs for MOX increased sharply at low concentration. This could be attributed to massive active sites which were readily accessible. Then, the increasing trend became slow with further increase of initial concentration, which indicated that there were less available active sites at the end of the adsorptive process. In order to study the mechanism of the adsorption, equilibrium adsorption data of the MOX were described using well-known HM, FM and LM. The correlation coefficients (R^2) obtained in fitting adsorption data in three models, the adsorption parameters (K_D, K_F, K_L and 1/n) for the three MOX onto the SCGs are referred in Table 1. The data showed that the HM model is the best one to interpret the absorption of MOX onto SCGs. Moreover, the fitted curve of the three models were shown in Figure 4. A non-linear LM can be used to describe homogeneous adsorption systems in which adsorption takes place on a homogeneous surface by a monolayer without any interaction between the adsorbed molecules. A Langmuir-type isotherm indicates that the compound has a moderately high affinity for the adsorbents at the initial stage of adsorption, whereas successively, it has increasing difficulty in finding vacant sites, finally reaching a maximum of adsorption. A non-linear FM with 1/n < 1 (L-shaped) indicates that the compound has a high affinity for the SCGs, and that adsorption occurs rapidly in the first stage; successively, it decreases as adsorption sites are filled, but never reaches saturation (Sheshmani*et al.*,2014; Wang *et al.*, 2017). The results shown in Figure 3 indicated that the curve of MOX fitted well to the HM. At the initial stage of adsorption, numbers of vacant sites were supplied for MOX, therefore, the curve increased sharply; however, adsorption weakened when the vacant sites were less. Moreover, the results were also verified by the data in Table 1.

CONCLUSION

In conclusion, a green, environmental-friendly adsorbent was supplied to the MOX adsorption. The solution pH and the SCGs amount had a great effect on the adsorption efficiency. The data showed that when the solution pH was 6 and the SCGs amount was 40 mg, the adsorption is strong. Also, the adsorption isotherm indicated that the Henry model fitted better than LM and FM. As a highly efficient adsorbent for MOX, SCGs could be a candidate to adsorb contaminants in environment in the future.

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