



## ADSORPTION OF Cu(II) IONS FROM SYNTHETIC SOLUTION BY SUNFLOWER SEED HUSKS

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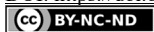
*Annual production of the sunflower seed in Serbia is between 650,000-720,000 tons. Most of this amount is used in vegetable oil industry. Abundant by-products from this processing are sunflower seed husks. Husks are usually incinerated by vegetable oil producers in order to obtain energy, used as an animal feed, or, unfortunately, landed up at some landfills. In order to promote new, added value for this abundant, renewable resource, the investigation presented in this paper was conducted. For that purpose, adsorption of Cu(II) ions from synthetic solution by unmodified sunflower seed husks was examined. ATR-FTIR was used to identify functional groups as potential active sites for Cu(II) sorption. Zeta potential values were determined to reveal the surface charge, while the cation exchange capacity (CEC) was determined to reveal the amount of exchangeable ions on its surface. ATR-FTIR analysis revealed the presence of specific functional groups (hydroxyl, carboxyl, carbonyl, and amine) responsible for removal of Cu(II) ions. The total CEC of sunflower husk is 47.74 meq/100g and Ca(II) and Mg(II) ions are in dominant exchangeable positions. The study of ion-exchange mechanism involvement was done and results confirmed that this mechanism is not the only mechanism which is involved in copper sorption. Also, the results show that the Cu(II) ions have preference for Mg(II) ions substitution. Sorption experiments were conducted in batch system. The effect of operating parameters (pH, contact time, initial concentration of Cu(II) ions and adsorbent dosage) on the adsorption capacity were investigated. The obtained experimental data were fitted by Langmuir and Freundlich isotherm models. The maximum adsorption capacity for Cu(II) ions calculated from Langmuir adsorption isotherm was 34.89 mg/g which is 15 to 35% higher than the capacity that other researchers reported previously for the same material and pollutant. These results are suggesting that sunflower seed husks have a potential to be applied as an effective adsorbent of copper ions from contaminated waters.*

**Keywords:** sunflower seed husks, copper, adsorption

### INTRODUCTION

Sunflower (*Helianthus annuus* L.) is one of the most important oil plants worldwide. Annual production of sunflower seed in Serbia is between 650,000-720,000 tons. Almost 50% of sunflower seed weight is waste (1), meaning that sunflower oil industry in Serbia each year generates approximately between 325,000 and 360,000 t of the waste sunflower husk. After industrial processing, this type of waste often ends up at landfills, or being used as energy source, or as an animal feed. World Health Organization has targeted copper as one of the most toxic metals and most common pollutants found in environment. In Serbia, one of the major environmental hot spots is the Serbian biggest mining industry, former Mining and Smelting Complex Bor (RTB Bor) which generates wastewater with a high concentration of copper (2). According to Milićević (3) whose work was focused on sorption of copper from Bor wastewater, it is necessary to develop the alternative methods and sorbents to reduce copper level to the 1 mg/L. Biosorption is one

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of applicable alternative methods, because it uses cheap, available and abundant, waste materials as biosorbents for pollutants removal from aqueous solutions (especially from diluted solutions where copper concentration is in the range 10-100 mg/L) (2). In order to add value to sunflower husk (SF), its application for copper removal from aquatic systems was investigated. Sunflower husk was previously examined for removal of different pollutants such as dyes and heavy metals. Oguntimein et al., (4) confirmed that raw sunflower hull has a potential to be used as a biosorbent material for the removal of Azure A chloride dye. The same authors examined acid treated sunflower husk for removal of textile effluent dye and it was very successful also (5). Mohammed (6) showed that raw sunflower shells have ability to remove Pb(II), Cd(II) and Zn(II) ions from aqueous solution. Paulovics and Bokányi (7) examined chemically modified sunflower hulls and results confirmed that this material has higher ability to reduce Pb(II) and Cd(II) ions concentration from aqueous solution. Removal of copper ions was examined by some authors: Feizi and Jalali (8) and Abdulhussein and Al wared (9) have shown that sunflower husk has a very good binding affinity toward copper ions. However in our study obtained sorption capacity with the same sorbent and same metal was slightly higher (by 15 and 35%, respectively).

## METHODS AND MATERIALS

### Biomass and copper solution preparation

Sunflower husks were obtained from company Dijamant, Zrenjanin. The sample was washed with deionized water to remove impurities and dried at 50 °C. Afterwards it was grinded in blade grinder (20,000-30,000 rpm). The obtained sample was labelled as SF. Initial copper solution was prepared by dissolving a precise amount of  $\text{Cu}(\text{NO}_3)_2 \times 3\text{H}_2\text{O}$  (analytical grade) in deionized water. Further, initial copper solution was diluted with deionized water to obtain the desired concentration for the purpose of experiments.

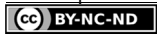
### Characterization of SF biomass

Attenuated Total Fourier Transform Infrared Spectroscopy (ATR-FTIR) was applied to determine surface functional groups before and after copper sorption, by using Thermo Nicolet 6700 FTIR. The zeta potential in the range from pH2 to pH10 was determined by using Zetasizer Nano Z (Malvern, U.K.). Cation exchange capacity (CEC) was determined by method with ammonium chloride (10).

### Adsorption experiments

Batch experiments were performed in 100 mL glass flasks containing 50 mL of copper solution. After adding sorbent SF in the solution, the mixture was put on thermostatic shaker at 250 rpm. After a specific period of time, the mixture was filtered and filtrate was analysed at atomic adsorption spectrophotometer AAS (Perkin Elmer AAnalyst 300). Operating parameters were obtained by investigating effect of pH, contact time, adsorbent concentration and adsorbate concentration. The effect of pH onto adsorption capacity was investigated in the range from 2 to 6. Effect of contact time was investigated in the range from 5 to 120 minutes. The effect of adsorbent concentration was examined in the range from 1 to 5 g/L, while effect of adsorbate concentration was in the range from 5 to 120 mg/L. From experimental data sorption capacities were calculated as:

$$q_e = \frac{V(C_i - C_f)}{m} \quad [1]$$



where  $q_e$  is equilibrium sorption capacity (mg/g),  $V$  is solution volume (L),  $C_i$  and  $C_f$  are initial and final concentrations before and after copper sorption and  $m$  is mass of SF (g).

In order to describe the sorption mechanism, obtained experimental data of sorption tests were subjected to equilibrium modelling by two commonly used two-parameter models (Langmuir and Freundlich). Table 1 summarizes Langmuir and Freundlich models used in this paper.

**Table 1** Equations and parameters of Langmuir (7) and Freundlich (8) models

Isotherm model	Equation	Parameter
Langmuir (11)	$q_e = \frac{q_{max}K_L C_e}{1 + K_L C_e}$ $R_L = \frac{1}{(1 + K_L C_0)}$	$q_e$ (mg/g): sorption capacity at equilibrium $q_{max}$ (mg/g): maximum sorption capacity $K_L$ (L/mg): Langmuir constant $C_e$ (mg/L): equilibrium concentration $R_L$ : dimension less separation factor $C_0$ (mg/L) - the highest initial metal concentration
Freundlich (12)	$q_e = K_f C_e^{1/n}$	$K_f$ (mg/g)(L/mg) <sup>1/n</sup> : Freundlich constant $n$ : heterogeneity factor

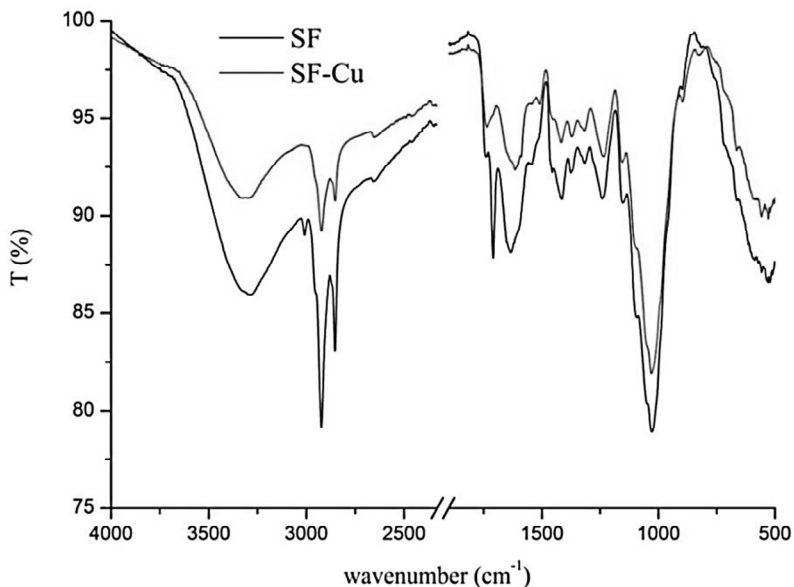
In the sunflower oil industry after de-hulling process of sunflower seed, the by-product sunflower husks (SF) are obtained. They consist of crude fibres (55.2%), crude proteins (5.7%), crude fat (4.6%) and fatty acids (3.5g/kg) with linoleic acid (22.6 g/kg) as a dominant fatty acid, followed by oleic acid (7.1 g/kg) palmitic acid (2.2 g/kg) and stearic acid (1.5 g/kg) (13). This lignocellulosic material consists mainly of cellulose (39%), hemicellulose (18%) and lignin (20%) (1). SF is also rich in mineral elements: calcium (4.1 g/kg), phosphorus (0.9 g/kg), magnesium (2.6 g/kg), sodium (0.09 g/kg), potassium (10.8 g/kg), sulphur (3.9 g/kg) (13). According to this, it can be concluded that SF contains chemical compounds that are able to bind heavy metals, due to the presence of specific functional groups (hydroxyl, carboxyl, carbonyl, amine and others (14).

## RESULTS AND DISCUSSION

### Characterization of SF biomass

#### ATR-FTIR

ATR-FTIR spectra analyses are significant in order to identify the functional groups on SF surface as potential active sites for copper binding. The ATR-FTIR spectra of SF before and after copper ions sorption are presented in Fig1. Since the SF is predominantly lignocellulosic material it consists of cellulose, lignin, and hemicellulose with its characteristic groups. The wide peak between 3100 and 3600  $\text{cm}^{-1}$  is assigned to the hydroxyl and amide groups, while peaks at 2923 and 2852  $\text{cm}^{-1}$  are assigned to asymmetric and symmetric  $-\text{CH}_2$  and  $-\text{CH}_3$  stretching vibrations (8). The very small peak at 3010  $\text{cm}^{-1}$  is assigned to C=C-H stretching vibration of fatty acids (15; 16). Peak at 1709  $\text{cm}^{-1}$  is assigned to C=O stretching of carbonyl group. Peak at 1027  $\text{cm}^{-1}$  could be assigned to  $-\text{C}-\text{OH}$  stretching vibration of alcohols and carboxylic acids (17).



**Figure 1** ATR-FTIR spectra before and after copper sorption onto SF

By comparing ATR-FTIR spectrum before and after sorption of copper ions onto SF, it is noticeable that more or less intensity of all bands decreased after metal sorption. The red-shift of the peak assigned to -OH and -NH group from 3331 to 3328  $\text{cm}^{-1}$ , indicate the involvement of these groups in the process of Cu(II) sorption. Also, the small peak at 3010  $\text{cm}^{-1}$  originated from fatty acids disappeared after copper ions sorption, indicating the involvement of carboxylic acids in metal bonding. The intensity of peak at 1709  $\text{cm}^{-1}$  assigned to C=O symmetric stretching, noticeably decreased showing the involvement of this group in copper bonding.

### **Cation exchange capacity (CEC)**

Cation exchange capacity (CEC) is the property of material to adsorb cations under chemically neutral conditions and it is reported in terms of milliequivalents of cations per 100 grams of material (18). The results of CEC are presented in Table 2.

**Table 2.** The cation exchange capacity of SF

Sorbent/cations	Na <sup>+</sup> (meq/100g)	K <sup>+</sup> (meq/100g)	Ca <sup>2+</sup> (meq/100g)	Mg <sup>2+</sup> (meq/100g)	Σ (meq/100g)
SF	2.16	6.65	23.45	15.47	47.74

The total CEC is expressed as a sum of alkaline and alkaline-earth ions (Na<sup>+</sup>, K<sup>+</sup>, Ca<sup>2+</sup> and Mg<sup>2+</sup>) which have been realised from SF, due to substitution with ammonium ions from the solution. The total CEC of SF is 47.74 meq/100g. It is observed that nearly 50% of all exchangeable ions are calcium ions (23.45 meq/100g), followed by nearly 30% magnesium ions (15.47 meq/100g), which means that those two are the dominant



ions in the exchangeable position. These ions could be exchanged with copper ions, by the ion exchange mechanism during the copper sorption onto SF.

### Zeta potential

Zeta potential values are negative all over the whole examined pH range, except at pH 2 where the electrokinetic potential is positive (+7.29 mV). The negative electrokinetic potential in pH range from 3 to 10 is -2.68 to -43.7mV, respectively. Negative zeta potential values indicate that SF has negatively charged surface functional groups. Therefore, SF may have exchangeable cations on its surface (19). This is in accordance with results obtained by FTIR and CEC analysis.

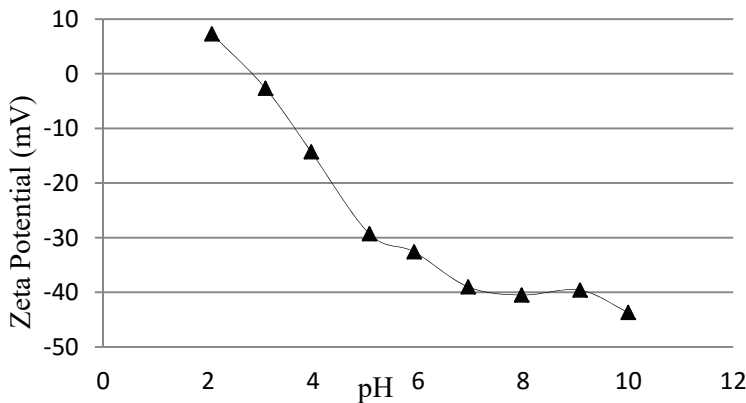


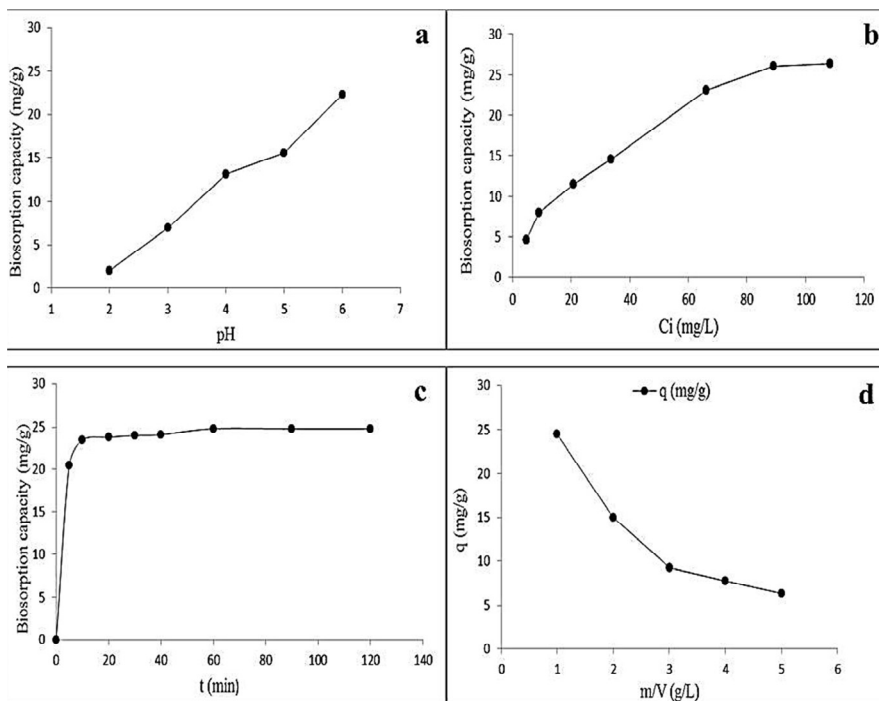
Figure 2 The zeta potential values of SF under various pH values range

### Operating parameters

**The effect of pH** on to sorption capacity has been investigated and the results are presented in Fig 3a. As can be seen from Figure 3a the SF has the lowest sorption capacity at pH2 (2.1 mg/g) and it slowly rises together with increase of pH value. When pH value is low the protons competes with copper ions for sorption sites at SF, resulting in low sorption capacity (20). As the pH value of the solution raises the copper ions have tendency to substitute the protons, which results in higher sorption capacity of SF toward copper ions. However, at pH value larger than pH 5 the process of copper hydroxide formation occurs. Since both processes (sorption and precipitation) are happening at the same time the value of sorption capacity is higher at pH 6, due to the false positive results. In this study the optimal pH value for copper sorption was chosen at pH5. **The effect of initial metal concentration** onto sorption capacity of SF was investigated in range from 5 to 120 mg/L and the results are presented in Figure 3b. The sorption capacity rises from 4.62 mg/g, at initial concentration of 5 mg/L, to 26.4 mg/g at initial concentration of 100 mg/L. It is evident that the enhancement of initial solution concentration is a mighty driven force to overcome mass transfer resistance of copper ions between solid and liquid phase (21). **The effect of contact time** between sorbent SF and metal solution is presented in Figure 3c. As can be seen from this graph, two phases have been observed: first very rapid phase (up to 10 minutes), followed by the second phase where equilibrium has been reached. The first phase of the process of copper sorption onto SF is very intensive due to the large concentration gradient and due to the large numbers of available active sites on surface. After that very small changes in the sorption



capacity are observed due to saturation of surface sites, and very slow diffusion of solution into the interior of SF (22).

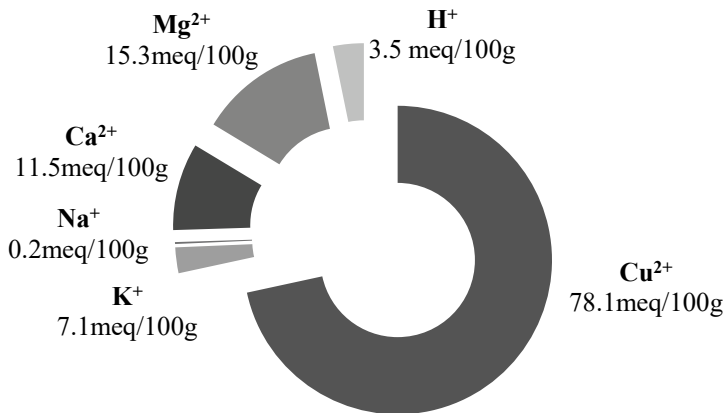


**Figure 3** Effect of pH value (a), initial concentration (b), contact time (c) and sorbent concentration (d) on sorption capacity of SF towards copper ions

The results of **the effect of sorbent concentration** on the sorption capacity are given in Figure 3d. As can be seen from the graph, the amount of adsorbed metal ions onto SF decreased from 24.5 mg/g to 6.34 mg/g as the amount of sorbent concentration increased from 1 to 5 g/L. The increase of sorbent dosage often induces particle aggregation and mixing difficulties, which can decrease effective surface area and active sites and slow down the mass transfer.

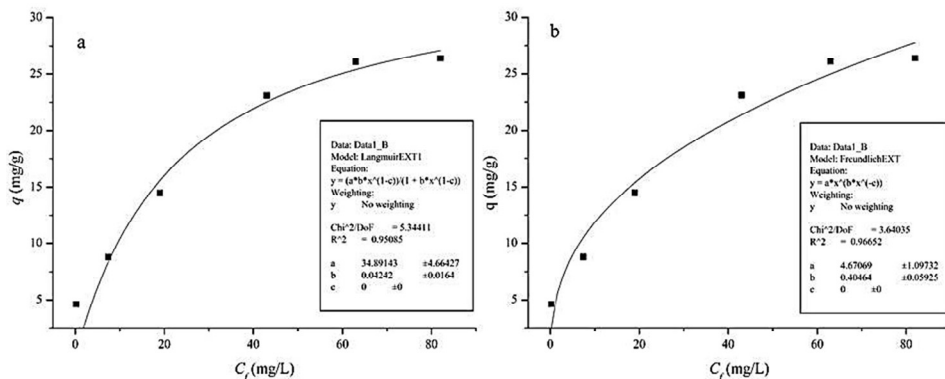
### Study of sorption mechanism

Since CEC analyses revealed that SF has a significant amount of cations in the exchangeable positions; the involvement of ion-exchange mechanism in sorption process is expected. For this purpose, the authors followed release of the exchangeable cations from SF during the process of copper sorption. The change of pH value was followed, too. The experimental parameters for investigation of ion exchange mechanism were: sorbent concentration was 1g/L; initial copper concentration was 60 mg/L; pH 5 and contact time: 2 h.



**Figure 4** Ion exchange mechanism vs copper biosorption

Figure 4 revealed that at this initial concentration the amount of released cations is 34.6 meq/100g, in comparison with the amount of bonded copper ions which is 78.1 meq/100g, which indicates that some other mechanism except ion exchange mechanism is involved in copper sorption process, such as electrostatic interaction with surface. Also, CEC analysis confirmed that dominant ions in the exchangeable position are  $\text{Ca}^{2+}$  (23.45 meq/100g) and  $\text{Mg}^{2+}$  (15.47 meq/100g). But after sorption experiment only 11.5 meq/100g (50% of amount calculated from CEC analysis) of calcium and 15.3 meq/100g of magnesium ions (all magnesium ions in exchangeable positions) were released from SF. According to Hillel (18) valences, radii and hydration properties of cations determine the preferences of those cations to be more or less readily exchangeable with metal ions. The smaller the ionic radius and the greater the valence, the more strongly the ion is bonded. Also, the greater the ion's hydration the bond is weaker between surface and that ion. Ionic radius of copper is 1.4 Å, magnesium 1.73 Å and calcium is 2.31 Å. Considering the fact that magnesium ions have smaller ionic radius than calcium, while calcium ions also have greater ionic radius than copper ions, copper ions will more readily exchange the magnesium ions than calcium ions from SF during the process of sorption. For a better understanding of sorption mechanism, experimental data were fitted by two-parameter common models: Langmuir and Freundlich (equations of models are listed in Table 1). The Langmuir model (11) presumes that the sorbent surface is homogeneous and the sorption occurs in monolayer without any interaction between sorbed ions. The Freundlich model presumes that the sorbent surface is heterogeneous and that the sorption occurs through multilayers due to interactions among sorbed ions (12). The isotherm parameters were: initial copper concentration from 5 to 120 mg/L, sorbent concentration of 1 g/L, value of pH 5, for period of 120 minutes, at room temperature and at stirring speed of 250 rpm. Calculated parameters from isotherm models are presented in Table 3.



**Figure 5** Langmuir (a) and Freundlich (b) isotherm models for the copper sorption

**Table 3** Isotherm parameters for Cu(II) onto SF

Modells	Parameters	SF	$R^2$
Langmuir	$q_m$ (mg/g)	34.89	0.951
	$K_L$ (L/mg)	0.04	
	$R_L$	0.17	
	$\chi^2$	5.34	
Freundlich	$K_f$ (mg/g)(L/mg) $1/n$	4.67	0.966
	$1/n$	0.40	
	$\chi^2$	3.64	

Considering the correlation coefficient  $R^2$  and chi-square value it is evident that the Freundlich model provides a better fit to experimental data, indicating that multilayer sorption occurs onto heterogeneous surface of SF. Since both isotherm models have similar values of  $R^2$  and due to the fact that Freundlich model can't show the maximum sorption capacity, one can consider that maximum sorption capacity is 34.9 mg/g from Langmuir isotherm model. Obtained sorption capacity is almost 15 to 35% higher than the capacity reported by other researchers (4; 5). According to Lopičić (2), the grinding process has to be very carefully designed, because inadequate grinding process may affect the porous structure of lignocelluloses materials, leading to clogging of the diffusion pathways and therefore affecting the sorption performance of material. The authors assumed that the reason for the higher sorption capacity of sunflower husks could be in a preparation procedure, in fact in a grinding process. Thanks to the blades from blade grinder which was used for sample preparation the material structure was preserved. The further investigations in this direction will be conducted.

## CONCLUSIONS

In this paper sorption of Cu(II) ions from synthetic solution by unmodified sunflower seed husks was examined. Negative zeta potential values indicate that SF has negatively charged surface functional groups and that it has exchangeable cations on its surface, which was confirmed by ATR-FTIR and CEC analysis. ATR-FTIR analysis revealed the presence of specific functional groups (such as hydroxyl, carboxyl, carbonyl and amine) on SF surface which are responsible for sequestering copper ions from aqueous solutions. CEC analysis revealed that the total CEC of SF is 47.74 meq/100g, with calcium





and magnesium in dominant exchangeable positions. However, study of involvement of ion exchange mechanism in copper sorption revealed that the copper ions have preference for magnesium ion substitution on SF surface. Due to the fact that magnesium ions have smaller ionic radius than calcium, copper ions will more readily exchange with the magnesium ions than with calcium ions from SF during the process of sorption. The sorption of copper ions onto SF occurs on heterogeneous surface in multilayers through interaction with other sorbed ions. The maximal adsorption capacity of SF for Cu(II) ions calculated from Langmuir adsorption isotherm is 34.89 mg/g, which is almost 15 to 35% higher than the capacity that other researchers presented previously. This brief study revealed that sunflower husks have a potential to be applied as an effective adsorbent of copper ions from contaminated waters. Since the sunflower husk is widely available as a by-product from oil industry, its application as sorbent is reasonable and sustainable.

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