

12-20-2015

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Recommended Citation

Sihombing, Riwardi; Krisnandi, Yuni Krisyuningsih; Widya, Rahma; Luthfiyah, Siti Zahrotul; and Yunarti, Rika Tri (2015) "Adsorption of Phosphate Ion in Water with Lithium-Intercalated Gibbsite," *Makara Journal of Science*: Vol. 19 : Iss. 4 , Article 1.

DOI: 10.7454/mss.v19i4.5166

Available at: <https://scholarhub.ui.ac.id/science/vol19/iss4/1>

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This work was partially funded by a UI-Cluster Research Grant No. 1712/H2.R12/HKP.05.00/2014.

Adsorption of Phosphate Ion in Water with Lithium-Intercalated Gibbsite

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Abstract

In order to enhance adsorption capacity of gibbsite ($\text{Al}(\text{OH})_3$) as an adsorbent for the adsorption of phosphate in water, gibbsite was modified through lithium-intercalation. The purification method of Tributh and Lagaly was applied prior to intercalation. The Li-Intercalation was prepared by the dispersion of gibbsite into LiCl solution for 24 hours. This intercalation formed an cationic clay with the structure of $[\text{LiAl}_2(\text{OH})_6]^+$ and exchangeable Cl^- anions in the gibbsite interlayer. A phosphate adsorption test using Lithium-intercalated gibbsite (LIG) resulted in optimum adsorption occurring at pH 4.5 with an adsorption capacity of 11.198 mg phosphate/g LIG which is equivalent with 1.04 wt% LIG. The adsorption capacity decreased with decreasing amounts of $\text{H}_2\text{PO}_4^-/\text{HPO}_4^-$ species in the solution. This study showed that LIG has potential as an adsorbent for phosphate in an aqueous solution with pH 4.5–9.5.

Abstract

Adsorpsi Ion Fosfat Dalam Air Menggunakan Gibsit Terinterkalasi Litium. Untuk meningkatkan kapasitas adsorpsi gibsit ($\text{Al}(\text{OH})_3$) sebagai adsorben untuk adsorpsi fosfat dalam air, gibsit dimodifikasi dengan cara interkalasi dengan litium. Gibsit dipurifikasi dengan menggunakan metode *Tributh and Lagaly* sebelum diinterkalasi. Gibsit terinterkalasi litium dibuat dengan mendispersikan gibsit ke dalam larutan LiCl selama 24 jam. Intercalasi ini menghasilkan tanah lempung kationik dengan struktur $[\text{LiAl}_2(\text{OH})_6]^+$ dan anion Cl^- yang dapat dipertukarkan di dalam interlayer gibsit. Pengujian adsorpsi fosfat menggunakan gibsit terinterkalasi litium (LIG) yang menghasilkan adsorpsi optimum terjadi pada pH 4.5 dengan kapasitas adsorpsi 11.198 mg fosfat/g LIG yang ekuivalen dengan 1.04 b/b% LIG. Kapasitas adsorpsi menurun dengan pengurangan jumlah spesi $\text{H}_2\text{PO}_4^-/\text{HPO}_4^-$ dalam larutan. Penelitian ini menunjukkan bahwa LIG memiliki potensi sebagai adsorben fosfat dalam larutan air dengan pH 4,5-9,5.

Keyword: adsorption, gibbsite, intercalation, lithium, phosphate

Introduction

The main cause of eutrophication is the existence of phosphate [1]. Industrial, agricultural, and domestic wastewater is a source of excessive phosphate in water. Exceeding 2 μM of phosphate ion in water could stimulate algal growth (eutrophication), which not only affects water quality but also kills aquatic life [2]. Therefore, excess phosphate in water should be removed using appropriate methods. One of the ways to reduce phosphate concentration in wastewater is by chemical precipitation. Calcium and aluminum ions are usually used as chemical agents to precipitate phosphate ions [3]. This method is only applicable with high

phosphate concentration due to the solubility of phosphate salt. Phosphate in waste water can only be discharged in limited amounts, which is 0.5–1.0 mg/L [4]. To obtain a low concentration of phosphate, alternative methods, such as adsorption and ion exchange, could be applied. Various types of materials have been used as adsorbents. Natural material is more desirable as an adsorbent because of its abundance on earth. In addition, natural material is cheaper than synthetic material. Various natural materials have been used as phosphate adsorbents, such as zeolite [5], clay [6], fly ash [7], red mud [8], and calcined eggshell [9]. The problem with using natural material is impurities [10]. Purification of material has to be done before

using the material as an adsorbent. The Tributh and Lagaly method has been used for the purification of natural material. This method has successfully reduced impurities in clay [11].

In this work, gibbsite was used as the source of natural material. Gibbsite is a mineral with the chemical formula of aluminum hydroxide $\text{Al}(\text{OH})_3$ and is the most common mineral in the soil environment [12]. Gibbsite is a mineral with octahedral layers, as shown in Figure 1. The layers in gibbsite consist of neutral charge; therefore, the bonds between the layers are van der Waals interaction, which is known to be weak [13]. The gibbsite structure is called dioctahedral because it is only 2/3 filled. Due to its hydroxide ions binding to two aluminums, the center 1/3 is empty. The octahedral holes could be used to insert small cations, such as lithium (Li^+ , $r = 0.6\text{\AA}$). The intercalation of Li^+ (introduced as the LiCl salt into solution) into the octahedral holes and leaving the Cl^- ions in the basal spacing will change the properties of Li-gibbsite, making it anionic clay [14]. Because of these properties, gibbsite has potential as an adsorbent for phosphate.

This study aimed to examine the adsorption behavior of phosphate ions on lithium-intercalated gibbsite prepared from Indonesian natural gibbsite under various pH conditions. The effect of the Tributh and Lagaly purification method that includes the purification of carbonate, iron oxide, and organic material was investigated in the phosphate adsorption behavior.

Materials and Methods

Material. The gibbsite used is from Bintan in Riau, Indonesia. The elemental composition of natural gibbsite is shown in Table 1. The chunks of gibbsite were crushed using a mortar and then sieved with a 100- μm stainless steel sieve. Then, the material was dried in the oven at 105 °C and labeled as natural gibbsite (NG) process, 25 g of gibbsite was dispersed in 50 mL of sodium acetate buffer pH 4.9. The mixture was stirred until the CO_2 gas disappeared. For iron oxide purification, gibbsite-purified carbonate was dispersed into 50 mL of citrate buffer pH 8.3. The mixture was added to 4 g of sodium dithionite, stirred for 10 hr and then allowed to stand for three days. The mixture was centrifuged and the gained sediment was then treated by purification of organic material by using 100 mL of 0.1 N sodium acetate and then added to 30 mL of 30% H_2O_2 . The suspension was stirred for 2 h at 90 °C and allowed to stand at room temperature for three days and then centrifuged. After that, the precipitate was dried at 105 °C for 2 h. This resulting material was labeled as purified gibbsite (PG) and then characterized by XRD and EDX.

For lithium intercalation, 100 g of PG was dispersed in 100 mL solution of 2 M LiCl . Then, the suspension was

Table 1. Elemental Composition of Natural Gibbsite

Element	%Wt
O	48.63
Al	31.41
Si	5.56
Ba	1.32
Fe	12.67
Co	0.40

stirred and heated at 90 °C for 24 hr. The suspension was centrifuged and the solid was washed with ice water until it was free of Cl^- in the filtrate. Furthermore, the solid was dried at 90 °C for 24 hr. This resulting material was labeled as lithium-intercalated gibbsite (LIG). This material was characterized using XRD and EDX.

Chemical. All chemicals (purchased from Merck) were used without further purification unless otherwise indicated. The sodium dihydrogen phosphate ($\text{NaH}_2\text{PO}_4 \cdot 2\text{H}_2\text{O}$), sodium phosphate dibasic (Na_2HPO_4), Ammonium molybdate ($(\text{NH}_4)_2\text{MoO}_4$), hydrogen peroxide (H_2O_2), lithium chloride (LiCl), hydrogen chloride (HCl), sodium citrate ($\text{C}_6\text{H}_5\text{Na}_3\text{O}_7 \cdot 2\text{H}_2\text{O}$), sulfuric acid (H_2SO_4), nitric acid (HNO_3), antimony potassium tartrate $\text{K}_2\text{Sb}_2(\text{C}_4\text{H}_2\text{O}_6)_2$, and ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$).

Adsorption experiment. Approximately 0.3 g of NG, PG, and LIG were used for each adsorption experiment and dispersed into 25 mL of 100 ppm of phosphate solution in pH 2.5, 4.5, and 9.5. Then the mixture was stirred for 5, 15, 30, 60, 120, 180, 240, 360, and 900 min. Subsequently, the mixture was centrifuged at 6000 rpm for three min and decanted. The phosphate concentration in filtrate was measured by using the molybdenum blue method, and the absorbance was measured by Shimadzu UV- Vis 4250. The effect of concentration was only performed for LIG in the range of the phosphate concentration of 5 ppm to 120 ppm. The adsorption isotherms were performed for 15 min.

Adsorption capacity was calculated using this equation:

$$Q = \frac{(c_i - c_s) \times V}{m} \quad (1)$$

Q = Adsorption capacity (mg P/ g Gibbsite)

c_i = initial phosphate (mg/L)

c_s = concentration after t minute adsorption (mg/L)

V = Volume solution (L)

m = weight of gibbsite (g)

Results and Discussion

Preparation and purification of gibbsite. The XRD patterns of gibbsite before and after treatment are shown

in Figure 1. Figure 1a shows the XRD pattern for natural gibbsite (NG). This XRD pattern shows the characteristic peaks of the mineral gibbsite at $2\theta=18.28$, 20.28 , and 37.7 [15]. Figures 1a and 1b from NG and PG, respectively, show that there is no significant change in XRD pattern before and after purification treatment, which indicates that the structure of gibbsite remains after being purified.

The results of EDX (Figure 2) show that other elements, such as Si, Ba, Fe, and Co, also exist in the gibbsite from Bintan, Riau. It can be seen that the iron content decreased significantly from 12.34 wt% in NG to 4.22 wt% in LiG. This result showed iron purification was successfully carried out.

Intercalation of lithium. The intercalation of lithium into the mineral gibbsite is expected to enhance the adsorption capacity of the mineral gibbsite. This method was conducted by dispersing gibbsite into a LiCl solution, which formed $[\text{LiAl}_2(\text{OH})_6]\text{Cl}\cdot x\text{H}_2\text{O}$. As explained earlier by Wang *et al.* [15], in the process of intercalation, Li^+ cations fill the octahedral holes in a layer of $\text{Al}(\text{OH})_3$ and Cl^- anions fill the position between the layers of mineral gibbsite.

The XRD analysis was applied to the prepared material in this study. Based on the literature, peaks at $2\theta=11.56$ and 23.2 are the typical peak of the Li^+ [14]. Figure 1c shows the XRD pattern of prepared LiG. It indicates

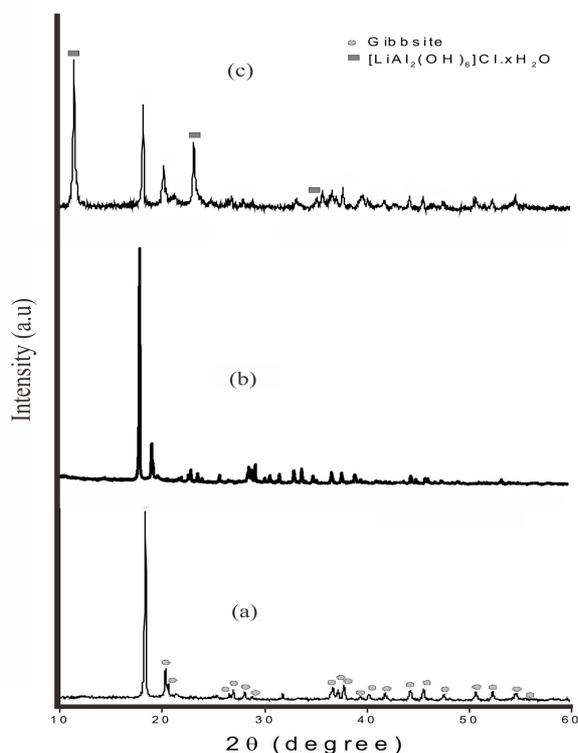


Figure 1. XRD Pattern of (a) Natural, (b) Purified, and (c) Lithium Intercalated Gibbsite

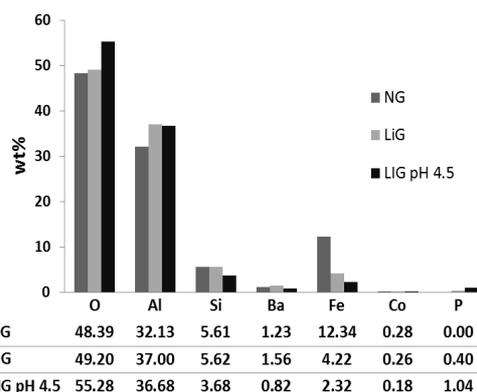


Figure 2. Summary of Element Concentration of Gibbsite Minerals before and After Treatment

that the peak of Li^+ ions appear after the intercalation process. A significant increase of basal spacing as the result of intercalation is not expected due to small size of Cl^- ions. The basal spacing of gibbsite is relatively unchanged—from 5.22\AA for PG to 5.23\AA for LiG. The existence of Li in the gibbsite structure is not detected by EDX due to the very high excitation energy ($K\alpha$) of Li.

Effect of adsorption time. The adsorption capacity of the three types of gibbsite, NG, PG, and LiG, were measured at 0–1000 min-adsorption time. The pH of solution chosen was pH 4.5, which is the condition in which the most abundant H_2PO_4^- is available in the solution [16].

Figure 3a shows the summary of results obtained at pH 4.5. It can be seen that NG adsorbed phosphate rapidly within 250 min and then slowed down, and it almost leveled after 1000 min adsorption. The phosphate uptake by PG is almost twice that of NG, but it reached a plateau shortly after 200 min adsorption. The adsorption uptake by LiG is the highest amongst the three gibbsite materials, almost three times of that by NG, and it kept going up even at 1000 min. The highest amount of phosphate adsorbed was noted when using LiG (7.65 mg/g) compared to 3.725 mg/g and 5.158 mg/g of NG and PG, respectively.

From this result, it is suggested that since the most abundant phosphate species that exists in pH 4.5 is H_2PO_4^- ion ($r = 2.38\text{\AA}$), it is feasible to exchange the H_2PO_4^- ion with Cl^- ion ($r = 1.67\text{\AA}$) inside the basal spacing of LiG ($0.4.8\text{\AA}$). The speed and the large adsorption capacity showed that the method of purification and intercalation carried great influence on the adsorption capacity of gibbsite.

Table 2 shows the comparison of adsorption capacity between LiG and another natural adsorbent. From this data, it shows that the adsorption capacity of LiG is potentially phosphate adsorbent.

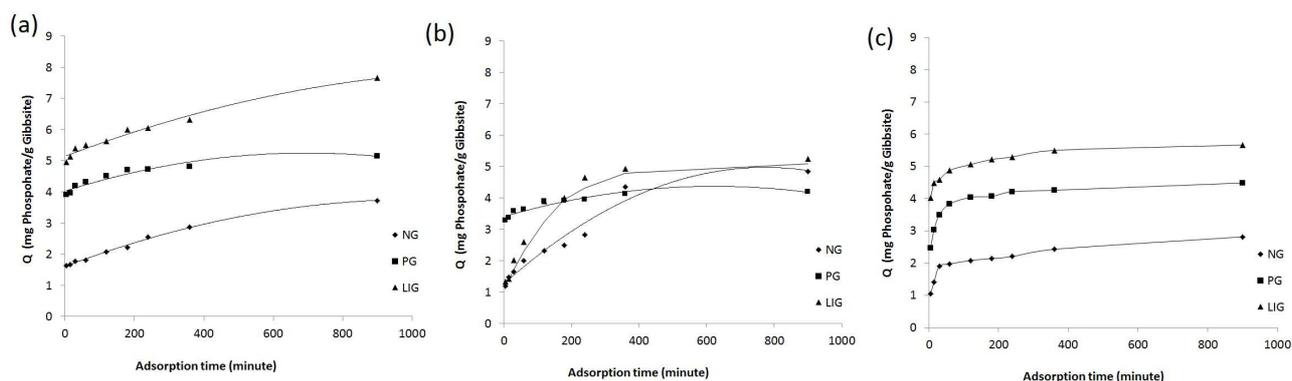


Figure 3. Phosphate Adsorption Capacity at pH: (a) 4.5, (b) 2.5, and (c) 9.5. Condition: 0.3 g Gibbsite, 25 mL of 100 ppm Phosphate Solution

Table 2. Comparison of Adsorption Capacity for Phosphate by Various Adsorbents

Adsorbent	Adsorption capacity (mg/g)	Ref
Calcined Eggshell	23.030	[9]
Al modified Clay	3.510	[6]
Red Mud	113.870	[8]
Fly ash	63.219	[7]
Li-Gibbsite (LIG)	11.198	This work*

*Result from Langmuir isotherm, Table 3.

Effect of initial pH. In order to observe the effect of the pH solution on the phosphate adsorption capacity of gibbsite materials, phosphate removal from aqueous solution in acidic (pH 2.5) and basic (pH 9.5) conditions were also carried out; the results are displayed in Figures 3b and 3c.

Figure 3b shows that the overall adsorption capacity for the three gibbsite materials at pH 2.5 is lower than at pH 4.5. The first phosphate uptake by NG and LiG was lower than that by PG. Furthermore, within 120 min, the phosphate adsorption capacity of NG and LiG increased rapidly and then remained constant for the rest of adsorption time. On the other hand, the adsorption capacity of PG was relatively constant throughout the observation period. After 360 min of adsorption time, the adsorption capacity was similar for NG and LiG and slightly lower for PG. This indicates that NG and LiG have more balance anion and cation compositions compared to PG. However, the three gibbsite materials could face structural damage due to the dealumination process at very low pH (2.5).

In terms of the phosphate species at pH 2.5, the H_3PO_4 species is in equilibrium with the $H_2PO_4^-$ species [16]. Thus, it is suggested that not all phosphate species can exchange the Cl^- ions inside the inter-gallery of gibbsite. This could decrease phosphate uptake by the gibbsite materials.

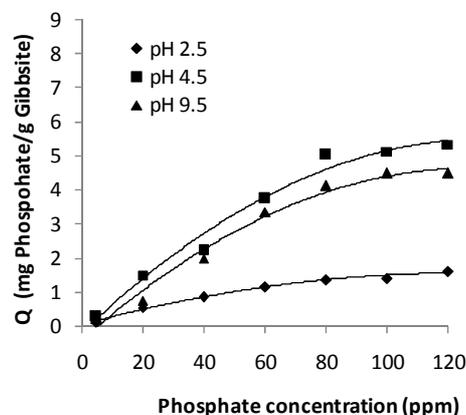


Figure 4. Phosphate Adsorption Capacity of LiG at Different pH

Figure 3c display the curves for the phosphate adsorption capacity of the gibbsite materials in a solution of pH 9.5, in which the abundant phosphate ion species is HPO_4^{2-} [16]. The HPO_4^{2-} anions could be exchanged with two chloride ions inside the LiG gallery, but the presence of other species, such as OH^- ions at alkaline pH would compete with phosphate and reduce the adsorption capacity.

Effect of the initial concentration. Effect of the initial concentration was conducted only with LiG. It can be seen from Figure 4 that at pH 4.5 and pH 9.5, the initial concentration of the phosphate solution affected the adsorption capacity, while at pH 2.5, the adsorption capacity only increased slightly when the initial phosphate concentration enlarged. Although the optimum Q value for LiG (5.5 mg/g) at pH 4.5 is higher than that observed at pH 9.5 (4.12 mg/g), the trend is not much different. The optimum Q value was obtained for an initial phosphate concentration of 80 ppm. This result indicates that the best condition for conducting phosphate adsorption is at pH 4.5, but the adsorption is plausible within the pH 4.5-9.5 range.

Further analysis of the results in Figure 4 was also conducted by plotting the results to Langmuir and Freundlich equations for isotherm adsorption.

Langmuir equation (2)

$$\frac{C_{eq}}{q_{eq}} = \frac{1}{q_m b} + \frac{C_{eq}}{q_m}$$

C_{eq} = equilibrium concentration of phosphate (ppm)

q_e = adsorbed phosphate at equilibrium (mg/g)

q_m = adsorption capacity (mg/g)

Freundlich equation (3)

$$\text{Log } q_{eq} = \text{Log } K_F + 1/n \text{Log } C_{eq}$$

C_{eq} = equilibrium concentration of phosphate (ppm)

q_e = adsorbed phosphate at equilibrium (mg/g)

K_F and $1/n$ = temperature-dependent constant for adsorbate and adsorbent

Figure 5 shows the plot for Langmuir isotherm adsorption; it can be seen that only adsorption at pH 2.5 follows the Langmuir isotherm adsorption ($R^2 = 0.9205$), while adsorption at both pH 4.5 and 9.5 give very poor R^2 . In the Langmuir model, it is assumed that at maximum coverage, there is only a monomolecular layer of phosphate species on the surface. This could be due to a neutral species of H_3PO_4 (the abundant phosphate species at pH 2.5), which is not favorable for exchange with Cl^- inside the inter-gallery of Li-gibbsite.

The plots of Freundlich isotherm adsorption are shown in Figure 6. It can be seen that although the adsorption at pH 2.5 gives good linearity (in order to obtain linearity, $R^2 = 0.9876$) the K_f is the lowest ($K_f = 0.1510$). In case of adsorption at pH 4.5 and 9.5, when the first two data are excluded, the R^2 are 0.6243 and 0.8563,

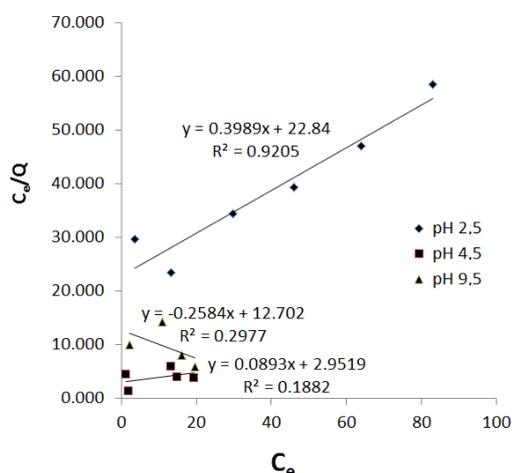


Figure 5. Langmuir Isotherm for Adsorption at pH 2.5, 4.5 and 9.5

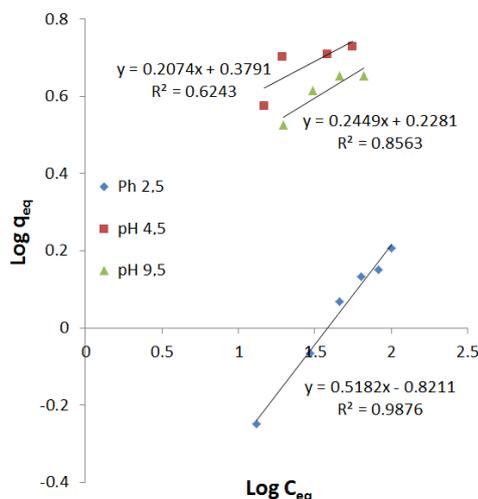


Figure 6. Freundlich Isotherm for Adsorption at pH 2.5, 4.5 and 9.5

Table 3. Summary of Analysis on Adsorption Isotherm for Phosphate Adsorbed in LIG

pH	Langmuir		Freundlich		
	Qm (mg/g)	R	Kf	1/n	R
2.5	2.5069	0.9205	0.1510	0.518	0.9876
4.5	11.198	0.1882	2.4952	0.244	0.6243
9.5	3.8670	0.2977	1.6908	0.207	0.8563

respectively, and the K_f improves, i.e., 2.4952 and 1.6908, respectively. Table 3 summarizes the analysis results. This suggests that the adsorptions fit the Freundlich isotherm adsorption only at higher initial phosphate concentrations, and pH 4.5 is confirmed to be the best pH condition for this phosphate adsorption. Furthermore, the phosphate adsorbed to LIG at pH 4.5 and 9.5 could create heterogeneous and multilayer stacking.

Conclusions

This study shows that the LIG is an effective and low cost adsorbent, which has a large available space for the removal of phosphate from aqueous solutions. Compared with non-intercalated and non-purified gibbsite, lithium-intercalated gibbsite is more effective as a phosphate adsorbent. In addition, the materials can be used in a wide range pH condition, i.e., 4.5–9.5, following the Freundlich isotherm adsorption model.

Acknowledgement

This work was partially funded by a UI-Cluster Research Grant No. 1712/H2.R12/HKP.05.00/2014.

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