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Simultaneous adsorption of lanthanum and yttrium from aqueous solution by durian rind biosorbent

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Abstract This paper presents the adsorption capacity of a biosorbent derived from the inner part of durian (*Durio zibethinus*) rinds, which are a low-cost and abundant agro-waste material. The durian rind sorbent has been successfully utilized to remove lanthanum (La) and yttrium (Y) ions from their binary aqueous solution. The effects of several adsorption parameters including contact time, pH, concentrations of La and Y, and temperature on the removal of La and Y ions were investigated. The adsorption isotherm and kinetics of the metal ions were also evaluated in detail. Both La and Y ions were efficiently adsorbed by the biosorbent with optimum adsorption capacity as high as 71 mg La and 35 mg Y per gram biosorbent, respectively. The simultaneous adsorption of La and Y ions follows Langmuir isotherm model, due to the favorable chelation and strong chemical interactions between the functional groups on the surface of the biosorbent and the metal ions. The addition of oxygen content after adsorption offers an interpretation that the rare-earth metal ions are chelated and incorporated most probably in the form of metal oxides. With such high adsorption capacity of La and Y ions, the durian rind sorbent could potentially be used to treat contaminated wastewater containing La and Y metal ions, as well as for separating and extracting rare-earth metal ions from crude minerals.

Keywords Simultaneous adsorption · Lanthanum · Yttrium · Biosorbent · Durian rind

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Introduction

Adsorbents derived from agricultural waste materials have the advantage of being natural, low-cost, non-toxic, biocompatible, and eco-friendly making them suitable for removal of many types of pollutants for environmental remediation and/or chemical separations (Omo-Okoro et al. 2018; Bhatnagar et al. 2015). When compared to their commercially available activated carbon counterparts, agro-waste–derived adsorbents have a large surface area, high adsorption capacity, and high reactivity (Omo-Okoro et al. 2018; Bhatnagar et al. 2015). Commercially available activated carbon is also not cost-effective for wastewater treatment due to its regeneration requirements (Omo-Okoro et al. 2018; Chandra et al. 2007).

Agrowastes for adsorbents is selected based on their availability, stability during storage, and easy activation process (Omo-Okoro et al. 2018). Durian (*Durio zibethinus*), one of the seasonal, tropical fruits grown in Southeast Asia (Foo and Hameed 2011; Hameed and Hakimi 2008; Penjumras et al. 2014), is only harvested once a year, creating a lot of surplus waste. The edible portion consists of only 15–30% of its overall weight (Wai et al. 2010); therefore, generating a lot of excess waste that is usually thrown away, burned, or sent out to landfills, creating major environmental problems (Penjumras et al. 2014; Wang et al. 2017).

Recently, durian waste materials have been shown to be a promising source of natural adsorbents (Wang et al. 2017; Ngabura et al. 2018). The adsorption capacity of the durian rinds may be attributed to their chemical content, specifically, cellulose (33%) (Penjumras et al. 2014), hemi-cellulose (15.5%) (Masrol et al. 2015), and pectin (10–15%) (Penjumras et al. 2014). Carbon aerogels derived from durian rinds possess basic and hydrophobic surface groups are porous and have a large surface area for removal of organic liquids (Wang et al. 2017). The utilization of durian rinds as adsorbents for the removal of pollutants could address the environmental and waste management issues; however, its usage has been sparsely reported (Ngabura et al. 2018). One interesting application for biosorbents derived from durian rinds is to separate and extract lanthanide metal ions from their crude minerals. The rare-earth metals are known to have carcinogenic effects and caused damage to cell membranes, affecting the human reproduction and the nervous systems (Hirano and Suzuki 1996), and therefore removing them from water systems by use of biosorbent may provide a possible solution to tackling this problem. What makes the durian rinds suitable are the presence of carboxyl, phosphate, hydroxyl, amino, and thiol functional groups for chelating the rare-earth elements (Fomina and Gadd 2014). Therefore, one may also consider that the presence of water-soluble polydispersed biopolymers contained in the durian rind might be effective to chelate the lanthanide metal ions (Wai et al. 2010; Hokputsa et al. 2004).

The pectin extracted from durian rinds has been previously demonstrated to adsorb lanthanum from aqueous solutions efficiently (Kusrini et al. 2018). In this study, the inner part of the durian rind is used as an adsorbent for the simultaneous removal of lanthanum (La) and yttrium (Y) ions from aqueous solution. The effects of adsorption parameters such as pH, initial concentrations of the rare-earth elements, temperature, and contact time on the removal of La and Y ions are evaluated in detail. This is a follow-up to ongoing research on utilization of low-cost adsorbents to remove rare-earth elements, which can be considered as a first step to isolate them from aqueous solutions by using biosorbents derived from durian waste materials.

Materials and methods

Materials

Lanthanum nitrate hexahydrate ($La(NO_3)_3.6H_2O$) was purchased from R&M Chemicals (Essex, UK). Yttrium nitrate hexahydrate ($Y(NO_3)_3.6H_2O$), hydrochloric acid (HCl 37%), sodium hydroxide (NaOH), and ethanol (EtOH) were purchased from Merck (Darmstadt, Germany). All chemicals were used as received without any further purifications. Stock solutions of La and Y were prepared separately by dissolving 1 g $La(NO_3)_3.6H_2O$ and $Y(NO_3)_3.6H_2O$ in 1 L distilled water, respectively, and they were mixed accordingly at later stages. Different La and Y concentrations were prepared by diluting the stock solutions with distilled water.

Preparation of adsorbent

Durian rinds were obtained from Depok, Indonesia. The outer bark of the durian rind was removed and the inner bark was cut into small pieces (ca. 1 cm), and it was dried in an oven at 60 °C for 24 h. The dried inner bark of the durian rind was grounded into a powder, with particle sizes less than 500 μm. This powder was stored in a sealed bottle prior to further experiments and characterizations.

Characterizations

Functional groups existing in the biosorbent derived from durian rind were characterized by Fourier-transform infrared (FTIR) spectrophotometer (Hitachi, Japan). Surface morphology and elemental composition of the biosorbent were evaluated from their field emission scanning electron microscopy-energy dispersive X-ray images (FESEM-EDX) (Hitachi, Japan). The concentrations of La^{3+} and Y^{3+} ions were determined using an X-ray fluorescence (XRF) analyzer (Rigaku, Japan).

Adsorption procedure

To test the adsorption capability of the biosorbent, 0.25 g of the durian rind sorbent was mixed with 100 mL of equimolar La and Y solution using a shaker operating at 200 rpm. The effects of adsorption parameters such as contact time, pH of the medium, and concentrations of La and Y were evaluated. The contact time was in the range of 0–120 min, and the concentration of La and Y was in between 80–160 and 58–116 g/L, respectively. The pH of the mixture was set to be between 4 to 6 by adding HCl into the mixture. The thermodynamic behavior of the adsorption was evaluated at temperatures between 30 and 60 °C.

Adsorption of the La and Y ions by the durian rind sorbent was calculated using $q_e = V(C_o - C_e)/M$, and its adsorption efficiency (η) was estimated using $\eta = (C_o - C_e)/C_o \times 100\%$. Here, q_e is the metal ions uptake (mg/g), V is the volume of solution (L), C_o and C_e are the initial and equilibrium concentration of La and Y (mg/L), respectively, which was determined by X-ray fluorescence (XRF) spectroscopy, and M is the weight of adsorbent.

Results and discussion

Morphology and elemental composition

Figure 1 shows the SEM images of the biosorbent derived from durian rind before and after adsorption of La and Y from the binary aqueous solution. The images indicate that the surface of the durian rind sorbent is uneven, rough, and porous (see Fig. 1a) with the existence of micron-sized grooves. The surface becomes smoother with reduced porous topology upon adsorption of La and Y metal ions. This suggests that the grooves on the surface of adsorbent are occupied after the adsorption process.

Based on EDX analyses, C, O, N, Al, and K were found to be the main elements present in the biosorbent, as shown in Table 1. Notably, the fresh adsorbent had three major components, namely, C (57.46%), O (29.23%), and N (6.21%), which are quite similar with the ultimate analysis of durian rind (C 60.31%, O 28.08%, and N 3.06%) that was reported by Chandra et al. (2007). In this study, both metals, namely, Al (1.07%) and K (5.39%), were also detected in the fresh durian rind. Upon adsorption of La and Y, the EDX analyses proved the existences of La and Y contained in the adsorbent. The level of La and Y metal ions adsorbed in the adsorbent was estimated to be 3.49 and 1.26%, respectively. This finding highlights the adsorption ability of the durian rind sorbent on the La and Y metal ions. Due to the incorporation of the La and Y metal ions on the surface of the sorbent, the elemental composition of the durian rind sorbent is modified.

It was also noted that the percentages of C and N decrease by at least 13.9% and O increases by 38.8%. The decrease in the percentage of C and N in the biosorbent could be due to either hydrolysis related to the leaching of soluble components from the biosorbent during adsorption, or the increase in mass due to the incorporation of La and Y metal ions into the durian rind sorbent. On the other hand, the increase in the percentage of O can be attributed to the incorporation of oxygen, along with the La and Y metal ions. This suggests that either the La and Y metal ions are chelated and incorporated in the biosorbent surface in the form of metal oxides or the biosorbent itself is oxidized during the drying process. It is noteworthy that the K and Al content in the biosorbent containing the La and Y metal ions is significantly decreased, up to 97 and 89%, respectively, which is much higher than the decrease in C and N elements. This significant decrease in the K and Al contents could be due to their removal from the durian rind sorbent, through an ion exchange process with the La and Y metal ions.

Functional groups

Figure 2 shows the FTIR spectra of the durian rind sorbent before and after the adsorption of La and Y metal ions. The main vibration bands of the durian rind sorbent before adsorption are at 1014 cm^{-1} due to C–O stretching, at 1600 and 1630 cm^{-1} attributed to C=O stretching of the carboxylate and carbonyl group, at 2961 cm^{-1} due to CH stretching, and a broad band peak at 3173 cm^{-1} assigned to OH and NH stretching (Wai et al. 2010). As shown in Fig. 2, the FTIR spectral pattern before and after adsorption were similar. The low-intensity vibrational bands originated from the La and Y metals due to their low loading level and low IR cross-section. The similarity in the spectral bands before and after adsorption also suggests that the functional groups on the biosorbent remain intact. This is a strong indication that the adsorption of the La and Y metal ions were due to the chelation process. This chelation is

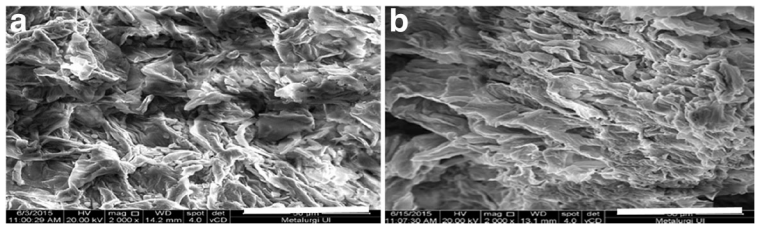

Fig. 1 SEM micrograph images of the durian rind sorbent; **a** before and **b** after La and Y adsorption from their binary aqueous solution. The scale bar in both images denotes 50 μm

facilitated by OH, NH, and CO functional groups which can be found on the surface of the durian rind sorbent. This notion is supported by the changes in relative vibrational intensity of the functional groups before and after adsorption.

The effect of contact time

The contact time between metal ions and sorbent is a key parameter for successful usage of biosorbent, especially in flow systems where rapid and practical removal is required. The optimum removal efficiency of La^{3+} and Y^{3+} ions approximately was 72 and 75%, respectively, at 120 min (see Fig. 3A), when equilibrium was attained. It is noteworthy that the adsorption rate depends on the presence of vacant adsorption sites and a high gradient concentration of solute (Gupta et al. 2014). Prior to the dynamic equilibrium, the adsorption of La and Y metal ions increases with contact time, indicative of the contribution of surface sites, and porous domains of the biosorbent. The rapid adsorption process is then followed by a slow process, and these two kinetic processes are common in many adsorption processes because the adsorbent contains high concentration of exchangeable binding sites with greater accessibility for the metal binding in the initial kinetic profile.

Table 1 EDX composition of the durian rind sorbent before and after the adsorption of La and Y from their binary aqueous solution

Before adsorption		After the adsorption	
Element	Weight (%)	Element	Wt.%
C	57.46	C	49.46
N	6.21	N	4.94
O	29.23	O	40.56
Al	1.07	Al	0.11
K	5.39	K	0.15
		La	3.49
		Y	1.26

The binding sites then become saturated and the rate of adsorptive uptake decreases with time.

The effect of initial concentrations of La^{3+} and Y^{3+}

The effect of the initial concentrations of La^{3+} and Y^{3+} on their adsorption efficiency is shown in Fig. 3B. Based on the XRF measurement, the optimum adsorption efficiency was 80.3 mg/g for La and 58.1 mg/g for Y. The removal efficiency for La and Y ions decreases nearly linearly with their initial concentration. The increase in the concentration of La and Y ions would limit the interaction between the metal ions and adsorbent due to saturation of the active site of the adsorbent. Thus, the optimum adsorption depends on the ratio between concentration of the metal ions and adsorbent dosage. In contrast with the current finding, adsorption of acid green 25 (AG25) by durian rinds in the presence of a mixture of acid red 88 (AR88), acid green 3 (AG3), and acid orange 7 (AO7) increases with the initial concentration of AG25 from 50 to 500 mg/L due to enhanced interactions between acid dye and durian rind (Hameed and Hakimi 2008). Similar effects were reported for adsorption of AR88, AG3, and AO7, using macroalgae

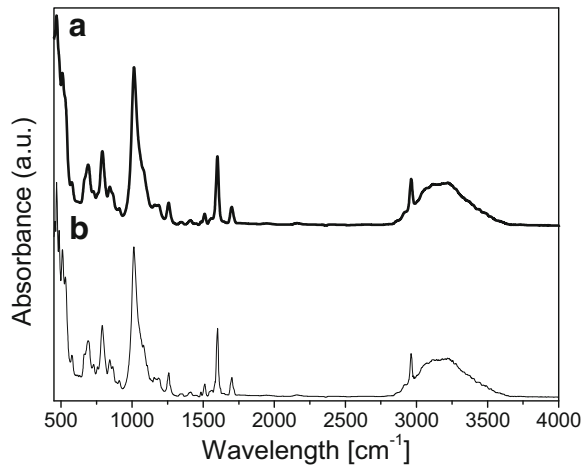

Fig. 2 FTIR spectra of the durian rind sorbent; (A) before and (B) after La and Y adsorption from their binary aqueous solution

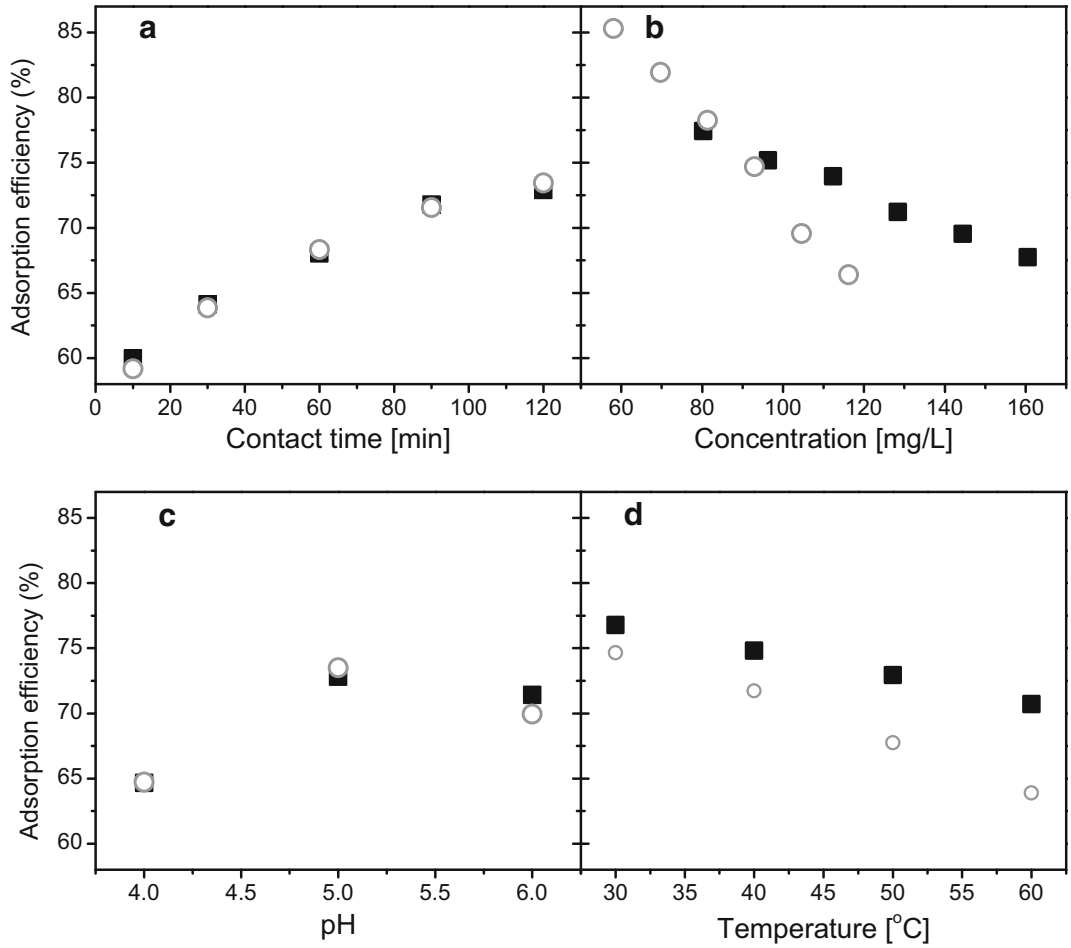

Fig. 3 The adsorption of La (black square) and Y (white circle) on the durian rind sorbent; the effects of (A) contact time, (B) initial concentration of La and Y, (C) pH of medium, and (D) temperature

Azolla filiculoides (Padmesh et al. 2005). These observations may be related to the additional effect of active sites on the surface of adsorbent and their relative binding affinity that differs from the durian rind sorbent.

The effect of pH

As shown in Fig. 3C, simultaneous adsorption of La and Y using the durian rind sorbent increased with pH in the range of 4–5, reaching optimum adsorption capacity (La 72.8% and Y 73.4%) at pH 5, and decreased at higher pH. In comparison, the optimum removal of La by pectin extracted from durian rind occurred at pH 4 (Kusrini et al. 2018). The pH-dependent adsorption capacity is attributed to the modification of the surface charge of the biosorbent in different environments, and the surface charge has been outlined to play a key role for the adsorption process (Foo and Hameed 2011). This suggests that functional groups on the surface of the biosorbent were negatively charged when pH of medium is higher than their dissociation constant (pKa) values. Considering that the pKa of carboxylate groups is in the range of 3.8–5, it is not so surprising that the dissociation of the carboxylate groups in the biosorbents was observed at pH 4–5, as indicated by the optimum adsorption of the La and Y ions.

At higher pH, above the pKa, for example, at pH 6, other dissociation equilibria and concentration anions such as hydroxyl ions are increased in the bulk solution. Consequently, La and Y ions can make hydroxyl complexes, changing their species into $[M(OH)_n]^{x-}$ (where M is the metal ions, n is an integer value, and x adopts a negative charge for $n > 3$ related to the offset electropositive character observed at low pH). In this condition,

the uptake of La and Y ions should decrease because the surface sites of the biosorbent are expected to adopt a negative zeta-potential at this high pH. The pH dependence of the La and Y ion adsorption has therefore been attributed to the ionization state of the binding site and the speciation of the adsorbate due to hydrolysis. These findings also indicate that the adsorption of La and Y ions by the durian rind sorbent relies on electrostatic interaction, in agreement with adsorption of cadmium ions and acid green dyes by durian rind (Hameed and Hakimi 2008; Saikaew and Kaewsarn 2009). The high adsorption efficiency of the La and Y ions may be facilitated by the extended surface area of the durian rind sorbent, as evidenced by the grooves and the active chelation groups on the surface of adsorbent. Similarly, removal of cadmium ions by pectin isolated from dried durian rind is most probably related to the same functional groups to those of the durian rind sorbent (Wang et al. 2016).

The effect of temperature

The effect of temperature, which indicates the thermodynamic behavior of the adsorption, was evaluated in the range of 30–60 °C. Figure 3 D shows the temperature-dependent removal efficiency of La and Y from the binary aqueous solution. It is clearly seen that the level of removal of both La and Y decreases with temperature, implying that the adsorption process is exothermic in nature, which is in agreement with the thermodynamic behavior of metal chelation in solution. In this study, the removal efficiency decreases at higher temperatures because the adsorption process is favored at lower temperatures. This can be attributed to the presence of carboxylate and hydroxyl functional groups of active sites on the biosorbent. It is anticipated that entropic effects due to hydration may play a role since the desolvation of the functional groups occurs more readily during metal ion chelation.

Adsorption isotherm

To evaluate the adsorption isotherm of the La and Y ion, two fundamental models, namely, Langmuir and Freundlich isotherm models, have been fitted to the experimental data. Both models relate the adsorption capacity (q_e) of the adsorbent and the equilibrium concentration (C_e) of adsorbate, as given by the two linear equations; $^1/_{Q_e} = (^{K_L}/_{Q_m})^1/_{C_e} + ^1/_{Q_m}$ and $\log Q_e = ^1/_n \log C_e + \log K_f$ for Langmuir and Freundlich isotherm model, respectively, (where n, K_L, and K_f are constants and Q_m is the monolayer adsorption capacity). In particular, based on the Langmuir adsorption model, the monolayer adsorption properties as given by $R_L = ^1/_{1+K_L C_0}$, (where C_0 is the initial concentration of the adsorbate), were also evaluated.

Figure 4 shows the linear plots of Langmuir and Freundlich isotherm models on the experimental data of the La^{3+} and Y^{3+} adsorption. The data fitted well with both the isotherm models, and from the correlation coefficient of the global fit on the experimental data, the adsorption of the La and Y ions on durian rind sorbent was best described by the Langmuir isotherm model.

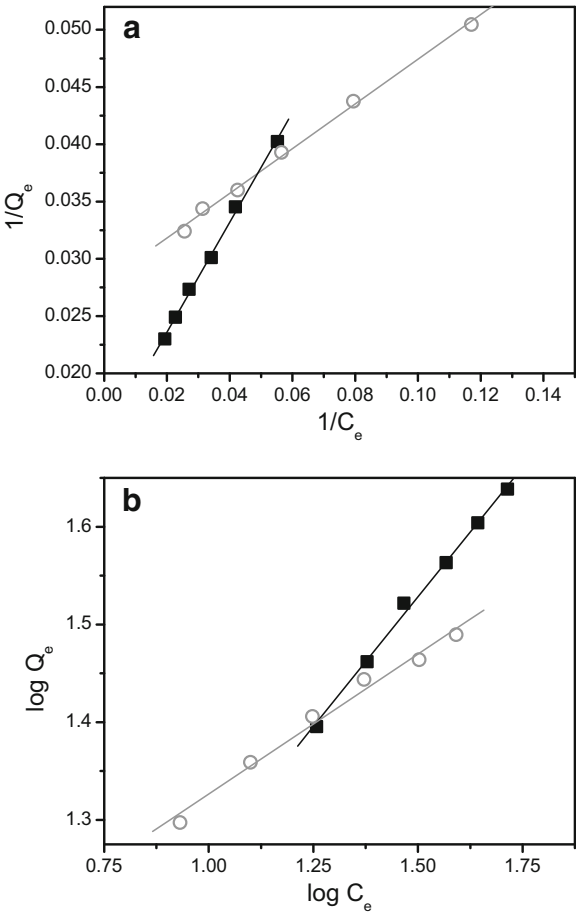

Fig. 4 Adsorption isotherm of La (black square) and Y (white circle) on the durian rind sorbent plotted by **a** Langmuir and **b** Freundlich isotherm models. The straight lines in both graphs are the best fits by Langmuir and Freundlich isotherm models

From the linear regression, the Q_m value was estimated to be 71 and 35 mg/g for La^{3+} and Y^{3+}, respectively, with K_L value being 38.8 and 6.8 for La^{3+} and Y^{3+}, respectively. On the other hand, the R_L value was estimated to be 0.0002 and 0.0015 for La and Y, respectively, indicating that the adsorption of La and Y ions is both favorable and irreversible. In comparison, the Q_m of the batch adsorption of zinc ions using durian rind sorbent is 36.73 mg/g (Ngabura et al. 2018) and that of La^{3+} adsorption using pectin extracted from durian rind is 41.2 mg/g (Kusrini et al. 2018).

Conclusion

In the present study, it has been demonstrated that the inner part of the durian rind can be utilized as an adsorbent for the simultaneous removal of La^{3+} and Y^{3+} from their binary aqueous solution, via adsorption process. By varying the contact time, pH of medium, and concentrations of the La and Y ions, the optimum adsorption capacity of La^{3+} and Y^{3+} was estimated to be 71 and 35 mg per gram biosorbent, respectively. The simultaneous adsorption of La and Y ions using the durian rind sorbent follows Langmuir isotherm model. The adsorption of La and Y ions was found to be exothermic in nature, which can be attributed to the chelation and strong chemical interactions between the functional groups on the surface of the biosorbent and the metal ions. Considering the high adsorption capacity of the durian rind sorbent for La and Y ions, this cost-effective biosorbent could potentially be used for treating contaminated wastewater, to remove rare-earth metal elements and also for their separation and extraction from crude minerals.

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