Synthesis of Mesoporous Materials from Vetiver Grass for Wastewater Treatment

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Abstract
In this work, mesoporous materials were synthesised by calcining vetiver leaves at 600°C for 3 hours in natural ambient. Afterwards, the particles were put in ultrasonic bath for 30 minutes and then dried before use. The chemical composition and morphology of the particles was examined using XRF and SEM, respectively. The performance of synthesised materials was evaluated using 10^{-5} M of methylene blue (MB) solution. The XRF results indicated the main composition of synthesised materials was ~45% of K. Others compositions were Si, P, Ca, Mg, S and Cl. The SEM images presented that synthesised materials was porous and less than 1 micron particles. After 10 minutes, ~95% of MB was removed using 3 g/L of mesoporous materials, which remained relatively stable after reusing the materials for 3 times. In summary, the present work shows that the leaves of vetiver grass can be used to synthesise mesoporous materials for wastewater treatment.

Keywords: vetiver grass, mesoporous materials, methylene blue.

INTRODUCTION

At the present time, the huge amount of wastewater discharged from various industries has led to an increasing demand of water purification. One of the most currently common treatment technologies is adsorption by porous materials [1,2]. With the desire of producing low-cost adsorbents, variety types of agricultural wastes including bamboo [1,2], sedge weed [3], pokeweed plants [4], sugarcane bagasse [5] have been used as a precursor to synthesise porous materials.

Vetiver grass is a tropical plant which origins in India [6,7]. *Vetiveria zizanioides (L.) Nash*, also known as *Chrysopogon zizanioides L. Roberty* is recognized as a commonly found species and can be found throughout the tropical and subtropical regions [8,9]. Vetiver grass can grow fast through tillering and usually appears in a dense clump. It has tall stems, thin and quite rigid leaves, and long roots. Its height can be up to 2 meter [10]. Two main utilisations of vetiver grass are erosion control, and oil extraction from its roots. On account of the massive and complex root system, vetiver grass is able to be applied as a low cost and effective solution for soil/water conservation and embankment stabilization [11,12]. In addition, the fragrant and volatile oil extracted from its roots has been used widely in various industries such as perfumery, cosmetics, soap, other fragrant materials production and medicine [13,14]. Recently, the application of vetiver grass in environmental treatment has been introduced, typically, phytoremediation due to its ability to accumulate and remove a variety of heavy metals, including manganese (Mn), iron (Fe), zinc (Zn), copper (Cu), arsenic (As), chromium (Cr), lead (Pb), cadmium (Cd) from contaminated soil and water [15,16]. Further, it has been reported that it can use to phytoremediate persistent organic pollutants (POPs) [10], phenol [17], and polycyclic aromatic hydrocarbons (PAHs) [18].

As above mentioned, it is clearly indicated that the vetiver grass has a high demand for industrial and environmental applications. However, it has to be noted that most of applications are about using vetiver roots. On the other hands, a large number of vetiver leaves are still free at the point of use and are discarded as agriculture wastes although a small portion of vetiver leaves is sometimes used for feeding cattle [19]. Thus, the intentions of the present work are to make a use for leaves of vetiver grass by synthesising mesoporous materials from

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vetiver leaves and use these mesoporous materials for wastewater treatment application.

METHODOLOGY

Synthesis and Characterisation

Vetiver grass was collected from the field in Phitsanulok Province, Thailand. Firstly, the vetiver leaves were cleaned with tap water and then were cut into small pieces. Afterwards, they were dried at 110°C for 12 h in ambient, this process was to remove moisture content. Then, the dried vetiver leaves were ground and sieved. The fraction with a particle size ranging from 0.1 to 0.2 mm was calcined at 600°C for 3 h using a muffle furnace in ambient condition, followed by natural cooling. The calcined vetiver particles were dispersed with deionised water by using an ultrasonic bath for 30 min. Finally, they were dried at 110°C and stored for use in dry cabinet. The chemical composition and crystal structure of the calcined vetiver particles was examined using X-Ray Fluorescence (XRF) and X-ray Diffraction (XRD), respectively. The morphology of the particles was obtained using Secondary Electrons Scanning Electron Microscopy (SEM).

Organic Pollutant Removal

The methylene blue (MB) removal efficiency of calcined vetiver particles was investigated using batch experiment at ambient condition. In a typical experiment, 0.15 g of calcined vetiver particles was placed into a 200 mL beaker containing 50 mL of 10^{-5} M MB solution. The mixture of calcined vetiver particles and MB solution was mixed by a magnetic stirrer for 10 minutes. The solid content was then removed using filter paper, and the efficiency of MB removal was achieved from measuring the absorbance of the leachate. The stabilisation of the calcined vetiver particles was examined by cleaning the used materials with deionised water in an ultrasonic bath for 30 minutes, and then reusing it. In the meantime, the MB removal efficiency of a type of commercial activated carbons (AC) was studied using the same procedure. The effect of different amount of calcined vetiver particles on MB removal efficiency was examined by varying the amount of the materials from 0.02 g to 0.2 g and then following the above steps.

RESULTS AND DISCUSSIONS

Characterisation of calcined vetiver materials

The XRF results indicate that the main chemical composition of calcined vetiver particles is potassium (K) with 45%, as shown in Table 1. Other compositions are 27% of silicon (Si), 11% of phosphorus (P) and small amount of other elements including calcium (Ca), magnesium (Mg), sulphur (S) and chlorine (Cl). Figure 1 shows the XRD patterns. It can be seen that the major phase of calcined vetiver particles could be potassium sulphate (K_2SO_4), according to JCPDS no. 00-005-0613. In addition, there are 2 minor phases which are silicon dioxide (SiO_2) and calcium phosphate (CaP), according to JCPDS no. 01-082-0512 and 00-016-0728 respectively. These data confirm the XRF results that the main composition of vetiver particles is potassium (K). These results are similar to the other investigation [19].

Table 1: Chemical compositions of calcined vetiver particles

No.	Tested Items	Tested Results	Unit
1	K	44.9	% w/w
2	Si	27.2	% w/w
3	P	10.7	% w/w
4	Ca	7.8	% w/w
5	S	3.7	% w/w
6	Mg	2.3	% w/w
7	Cl	2.1	% w/w
8	Fe	0.8	% w/w
9	Mn	0.5	% w/w

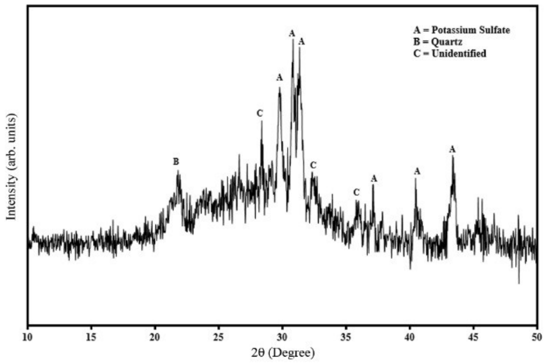

Fig. 1: XRD pattern of calcined vetiver particles

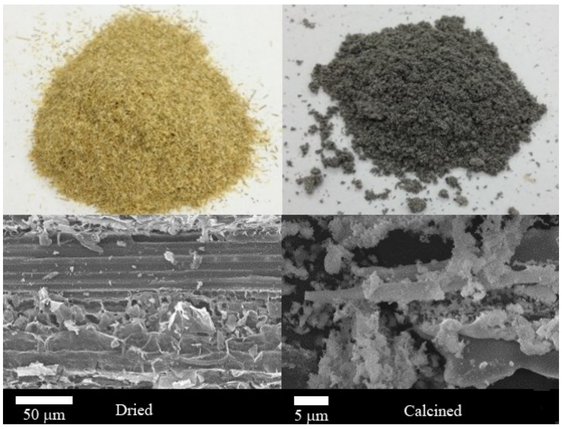

Fig. 2: SEM images of dried vetiver leaves and calcined vetiver particles

Figure 2 shows the SEM images of surface morphology of calcined vetiver particles. The images present that calcined vetiver particles are porous and fine particles. Before calcination process, the vetiver leaves are solid particle with 0.1-0.2 mm of size. However, after calcination at

600°C, the particles change to be fine particles with a size less than 1 micron. It can be said that calcination process can be used to synthesis fine particles with high porosity and surface area. The calcined vetiver particles can be called mesoporous materials.

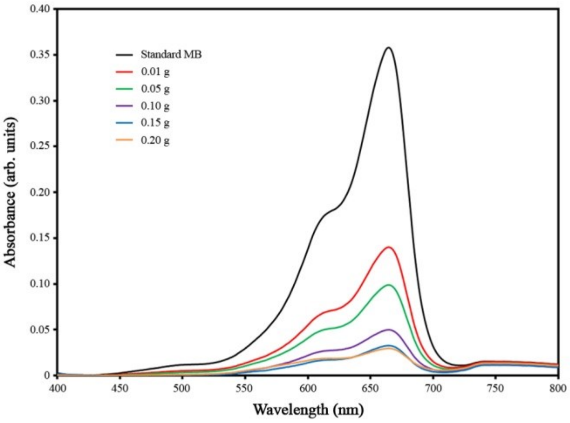

Fig. 3: MB absorbance with different amount of mesoporous materials

Efficiency of MB removal

The effect of the amount of mesoporous materials on MB removal efficiency is presented in Figure 3 and 4. It reveals that increase of amount of particles from 0.02 g to 0.2 g results in the decrease of the absorbance intensity of MB solution. This means the amount of particles directly affects the MB removal efficiency. The MB removal efficiency increased dramatically from 66% to 98%, as shown in Figure 4. It has to be noted that there is no significance in the MB removal efficiency between using 0.15 g and 0.2 g. Then, 0.15 g of mesoporous materials is used in the next experiments.

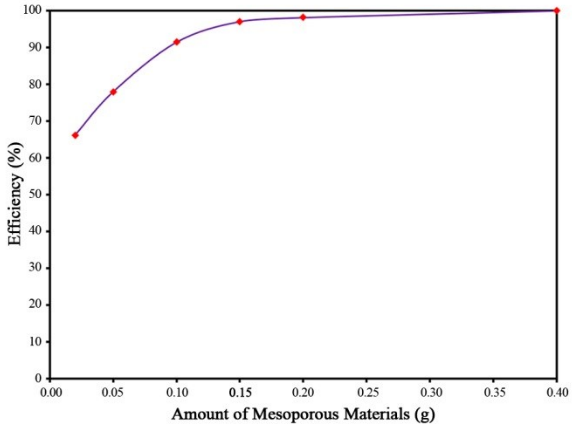

Fig. 4: MB removal efficiency with different amount of mesoporous materials

Figure 5 and 6 present the comparison of MB removal efficiency between mesoporous materials and commercial activated carbons. It proves that the mesoporous materials provide the higher MB removal efficiency, particularly 97%, than commercial activated carbons which can remove only 46% of MB. When reusing the mesoporous materials for three times, the absorbance of the treated MB solution fluctuated from 0.0172 to 0.0028, which means there was only a minor change in the range of 99% to 95% in term of MB removal efficiency. On the other hands, the treated MB solution absorbance when reusing commercial activated carbons for three times was 0.18, 0.2 and 0.24, respectively. This means the efficiency of commercial activated carbons significantly dropped from 46% to 29% after three times reuse. These results suggest that the mesoporous materials are very stable, easy to desorb in a simple way of using deionised water, and can be reused several times. However, it has to be noted that K_2SO_4 is soluble material. This property should results of weight loss during the recycle process.

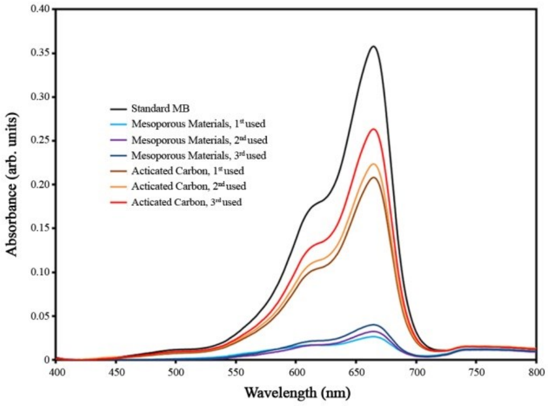

Fig. 5: MB absorbance of three times reusing mesoporous materials (V600) and commercial activated carbon (AC)

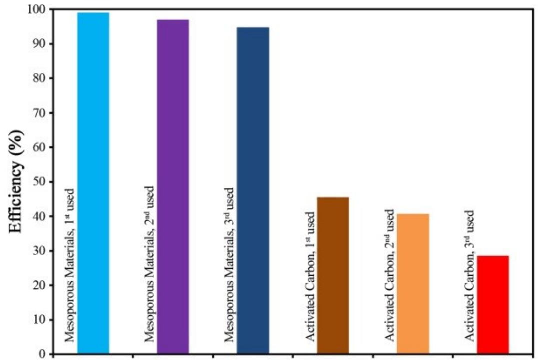

Fig. 6: MB removal efficiency of three times reusing mesoporous materials (V600) and commercial activated carbon (AC)

CONCLUSIONS

The present work has succeeded in synthesis of the mesoporous materials by calcining the leaves of vetiver grass at 600°C. The XRF data indicates that main chemical composition of mesoporous materials is potassium (K). In addition, minor compositions of mesoporous materials are (Si), phosphorus (P), and calcium (Ca), magnesium (Mg), sulphur (S) and chlorine (Cl). The XRD data indicates that main crystal structure of the mesoporous materials is potassium sulphate (K_2SO_4). The mesoporous materials are found to have the ability to remove MB with above 95% at a ratio of 0.15 g of the materials per 50 mL of MB solution. In addition, the materials were also proven to be quite stable, easy to desorb in a simple way of using deionised water, and can be reused many times with relatively steady performance. However, the effect of weight loss during the recycle process needs a further investigation. In conclusion, the present work suggests that the leaves of vetiver grass can be used to synthesise mesoporous materials that can be applied to wastewater treatment.

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