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Development of pellet-type adsorbent based on water treatment residual

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A B S T R A C T

To obtain a low-cost adsorbent with a regular shape for the better application in water pollution control practice, drinking water treatment residual (DWTR) was modified by adding different proportions of kaolin clay, and the pellet-type adsorbents were then made under the thermal treatment approach. Response surface methodology (RSM) was applied to optimize the production process and analyze the effects of firing time, firing temperature and DWTR proportion on phosphorus (P) adsorption capacity and mass loss ratio, which was chosen as the parameter of strength of the resultant DWTR-clay pellets. The results showed that an increasing temperature contributes to greater strength but leads to smaller P adsorption capacity, while kaolin clay addition results in an improvement of strength but a decrease of P adsorption capacity. The best P adsorption of 10.2 mg P/g pellet was obtained under the conditions of making the pellet with 40% clay addition while heating at 650°C for 60 min. The strength of DWTR pellet could be increased by thermal treatment with kaolin clay. This study can help the large-scale application of DWTR as a low-cost adsorbent in many kinds of water and environmental engineering practices, such as using DWTR as a substrate in the artificial floating island.

Keywords: Drinking water treatment residual; Modification; Phosphorus adsorption; Pellet-type adsorbent

1. Introduction

Drinking water treatment residual (DWTR) is a kind of inescapable by-product in water treatment processes. Great volumes of DWTR are generated worldwide due to the high demand for clean water [1]. As a result, reuse of DWTR has been gaining increased attention in recent years. With the high percentage of Al and/or Fe (oxy)hydroxides and the amorphous structure, DWTR is able to develop adsorbents for wastewater contaminants immobilization. These include phosphorus (P) [2], fluorides [3], perchlorate [4], chlorpyrifos [5], glyphosate [6], tetracycline [7], arsenic [8], selenium [9], etc. and heavy metals of Cd [10], Cr [11], Cu [12], Hg [13], Ni [14], Pb [15], Zn [16], etc. Recently, some approaches have been applied to DWTRs to modify their adsorption characteristics. For example, oxygen-limited heat treatment makes DWTR a more reliable adsorbent by increasing specific surface area and the sequestrated carbon [17]. Ultrasonic-assisted extraction and synthesis method turn DWTR to be an effective adsorbent for ammonium removal [18]. In addition, ultra-sonicated DWTR has an increased enrichment potential of weakly hydrophobic acid [19].

However, from a practical application point of view, the application of DWTRs as an adsorbent is still limited due to the low strength of "DWTR cake", which can be easily broken into pieces. As a result, shaping DWTR into various

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forms with proper strength received great attention from researchers. As such, the efforts have been mainly made in two aspects: thermal treatment [20] and adding binder [21]. It has been noted that little was done about creating DWTR-based adsorbent with special shape by thermal treatment. This study was aimed to investigate the possibility of developing shaped DWTR-based adsorbent by thermal treatment for the purpose to expand DWTR implementations.

Response surface methodology (RSM) is a multivariate statistical technique to explore and optimize the relationships between several explanatory variables and one or more response variables [22]. RSM had been developed and widely applied in many areas including water environment [23]. In this study, kaolin clay and thermal treatment were adopted to modify a DWTR, collected from the largest water treatment plant in Ireland, to obtain pellet-type adsorbent. RSM was employed to predict the optimized conditions to achieve high-performance in P adsorption capacity and strength. It is highly expected that this study will provide first-hand experience and understanding of the pellet-type DWTRs making, thus expanding the scope of their large-scale application as "a waste to product" in water pollution control.

2. Material and methods

2.1. Raw material

DWTR was obtained directly from the dewatering unit of the Ballymore Eustace Water Treatment Works Co., Kildare, Ireland, where the reservoir water was treated by dosing aluminium sulphate as the coagulant. The DWTR cake was "chocolate-like" cake in appearance with a moisture content of 78%–82%. After collection, the DWTR cakes were air-dried and then subjected to oven-drying at 105°C for 24 h. Chemical compositions were determined by inductively coupled plasma-atomic emission spectrometer, TOC-V CSH (Shimadzu, Beijing, China) and Fourier transform-infrared (EQUINOX-55), with 46% aluminum (as Al_2O_3), 1.2% iron (as Fe_2O_3), 1.1% calcium (as CaO), 0.7% magnesium (as MgO), 1.6% chlorine (as Cl^-), 0.1% sulfur (as SO_4^{2-}), 1.2% silicon (as SiO_4^{2-}), 10.2% H_2O (at 105°C as moisture content), 27% H_2O (at 1,000°C), 9.8% humic acid and other minor elements.

The kaolin clay was obtained from Sigma-Aldrich (Wicklow, Ireland) with linear formula of $Al_2Si_2O_5(OH)_4{\cdot}2H_2O$. There is little Fe (9.7%) and Pb (0.9%) in kaolin clay. The loss on ignition value is 12% at the standard temperature of 600°C.

2.2. Pellet adsorbent making

As shown in Fig. 1, the dried DWTRs were ground and sieved to a diameter of 0.063–0.150 mm. Kaolin clay, without further treatment, was added as the binder in different proportions to the prepared DWTR powder to shape pellets. Thereafter, deionized water was added into this DWTR/clay mixture with amount of 0.5 mL/g to keep moisture. This mixture was vigorously stirred at 5,000 rpm by a top driver blender (No. 7012 Townson & Mercer Ltd., Manchester, United Kingdom) for 1 h to obtain a homogeneous mouldable paste. The resultant mouldable paste was then introduced to a big needle with an inner diameter of 6 mm. The paste was extruded out of the needle and then cut to pellet with a length of 6 mm by a knife. As all the pellets are hand-made, the final diameter of adsorbent ranges from 5 to 8 mm. These pellets were then fired in a furnace at RSM designed temperature for the designed time with a 5°C/min speed in heating.

2.3. P adsorption

Batch tests were used to explore the equilibrium characteristics especially the P adsorption capacity of the resultant DWTR pellet-type adsorbent. The P solutions were prepared with deionized water and the pre-weighed potassium dihydrogen phosphate (KH_2PO_4). 1 g of the DWTR-clay pellet was equilibrated with 100 mL of P solution with concentrations ranging from 0 to 3,500 mg/L for 96 h on a shaker (SSL1, Bibby Sterilin Ltd., UK) at a speed of 200 rpm. After the set time (96 h), the mixture was withdrawn and filtered using a 0.45 μm Millipore membrane filter and analyzed for residual P using a HACH DR/2000 spectrophotometer according to its standard procedures. Thereafter, the P adsorption test was conducted and the adsorption behavior was described by the Langmuir isotherm.

2.4. Mass loss ratio

The mass loss ratio was used to evaluate the strength of pellets. Normally, compressive strength is a direct indicator of the strength of the material, but the small pellets do not fit the conditions of most press machines in a strength test. Instead, the mass loss ratio was utilized to predict the strength since it is significant to show the retention of structure in water for prospective application of pellets in the artificial floating island (AFI). To do so, the pellets were added to a 250 mL of distilled water and then shook for 124 h at 200 rpm. Thereafter, the pellets were dried for 24 h at 105°C. The difference in mass before and after was treated as mass loss ratio in Eq. (1). A small mass loss ratio means little change of the pellets during the shaking in water, indicating a great strength of pellets when pellets have been applied in water.

$$R = (m_0 - m)/m_0 \tag{1}$$

where R is loss ratio, m_0 is dry mass before the test (g) and m is dry mass after the test (g).

2.5. RSM

In the application of RSM, there are mainly three steps [23]: (1) design and experiments, which include selecting the independent variables and the delimitation of the experimental region, according to the previous studies and preliminary experiments; (2) response surface modelling through regression, which is mainly the data analysis process; (3) optimization and validation.

Previous studies suggested that thermal treatment of DWTR affected both P adsorption capacity [24] and comprehensive strength [25]. DWTR proportion in the DWTR/clay mixture played an important role in P adsorption due to the difference in P adsorption ability between DWTR and kaolin clay [26,27]. Additionally, comprehensive strength of DWTR-clay mixture can be affected by DWTR proportion in the mixture [28]. Therefore, parameters of thermal treatment (firing temperature and time) and DWTR proportion were

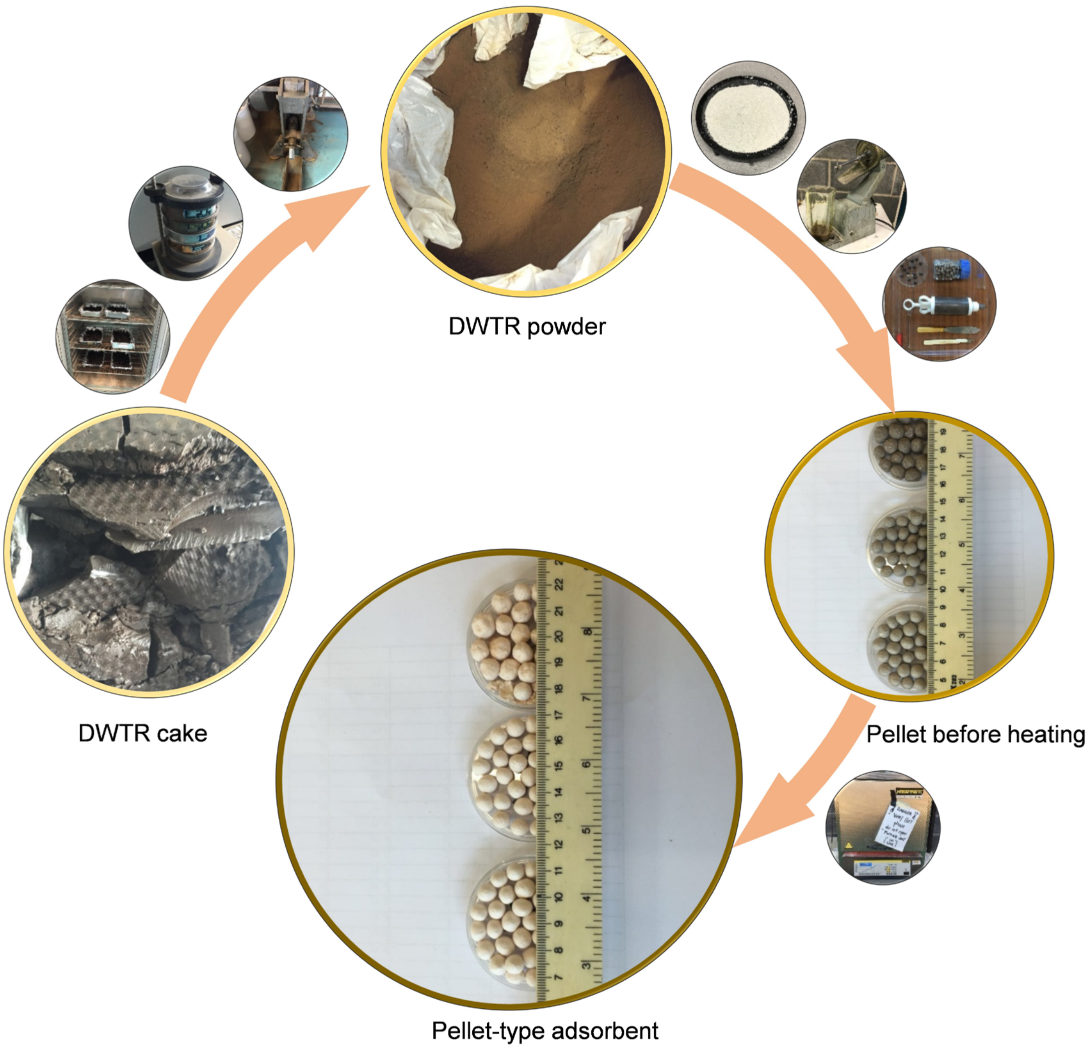

Fig. 1. Schematic description of pellet-type adsorbent making.

determined as three variables in RSM, as these three factors affect both P adsorption capacity and comprehensive strength.

Preliminary batch tests were employed to obtain the range of three factors in RSM. For a high comprehensive strength (low mass loss ratio) and a great P adsorption capacity, the temperature range seemed suitable from 600°C to 1,000°C. It was found that the pellet adsorbents could not remain in their pellet shape with a firing temperature of 600°C, while a significant reduction in P adsorption capacity occurred when the temperature is greater than 800°C (Fig. 2(a)). For a better P adsorption capacity, the DWTR proportion should be greater than 30%. However, it should not exceed 70% due to the fact that mass loss ratio increased sharply when DWTR proportion is higher than 70% (Fig. 2(b)). Regarding the firing time, no more positive influence on P adsorption and comprehensive strength were observed when the time is longer than 60 min (Fig. 2(c)). Thus, the range of firing temperature was set from 10 to 60 min for energetic and economic consideration. The RSM test linked with a number of P adsorption experiments to achieve maximum P adsorption capacity and minimum mass loss ratio was designed by a central composite design. The ranges and levels of the three variables/factors are presented in Table 1. In order to estimate the experimental variability and the experiment design, each factor was evaluated at high (+1), central (0) and low (–1) levels as cube points.

A total of 17 experimental sets were designed by RSM. All the experiments were carried out in triplicate and average values were taken as responses in specific operational conditions and used for further statistical analysis.

3. Results and discussion

3.1. RSM results towards the optimal condition of the pellet adsorbent making

The experimental results concerning P adsorption capacity and mass loss ratio are shown in Table 2. Here, P adsorption capacity is an important parameter of adsorption characteristics because mass loss ratio can be used to indicate

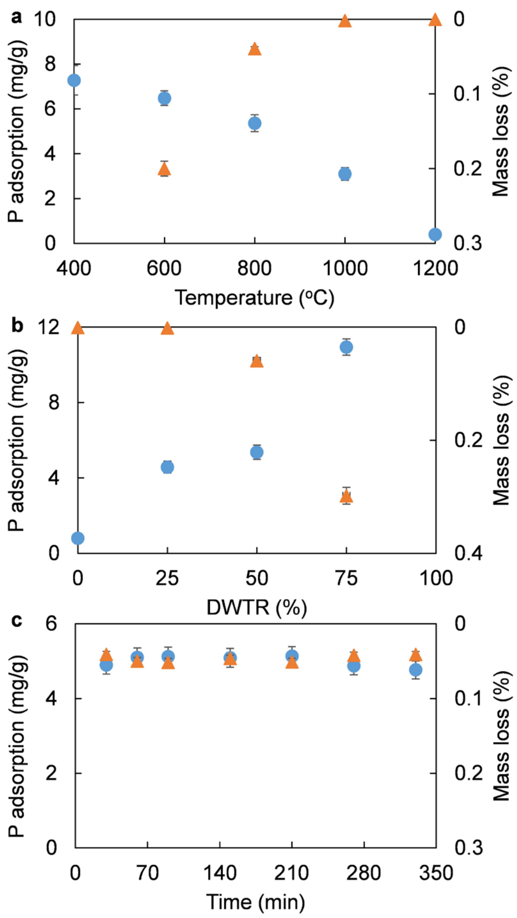

Fig. 2. One-factor tests for selecting range in RSM (P adsorption capacity, mass loss ratio, the adsorption conditions have been described in section 2.3).

Table 1
Ranges and levels of variables in DWTR-clay pellet making

Variable	Ranges and levels		
	–1	0	1
Firing time (min)	10	35	60
Firing temperature (°C)	600	800	1,000
DWTR proportion (%)	30	50	70

the strength of the material. Subsequently, these results were statistically analyzed and the P adsorption capacity and mass loss ratio were found to fit with second-order polynomial equations of Eqs. (2) and (3), respectively:

$$\text{Adsorption capacity} = 5.28 + 0.3A + 2.03B - 1.73C + 0.96AB - 0.38AC - 0.29BC + 0.50A^2 + 1.32B^2 - 0.78C^2 \quad (2)$$

$$\text{Mass loss ratio} = 0.056 - 0.00913A + 0.083B - 0.090C - 0.032AB - 0.011AC - 0.054BC + 0.025A^2 + 0.035B^2 + 0.013C^2 \quad (3)$$

The F-test for analysis of variance (ANOVA) was used to evaluate the significance of these two model equations. The central idea of ANOVA is to compare the variation due to the treatment (change in the combination of variable levels) with the variation due to random errors inherent to the measurements of the generated responses. From this comparison, it is possible to evaluate the significance of the regression used to foresee responses considering the sources of experimental variance. From the ANOVA results (Table 3), it is significant to use these two quadratic models to navigate the design space. The prob > F-values for models of both P adsorption capacity and mass loss ratio are lower than 0.05, indicating that they are desirable and significant models. By comparing residual error with a pure error from replicated design points, "lack of fit" values are greater than 0.05, demonstrating that there is no significant lack of fit and these two models could be used as response predictors. The coefficient of determination (R^2) that was found to be close to 1 (0.94 for adsorption capacity and 0.85 for mass loss ratio) also advocated a high correlation between observed and predicted values.

The three-dimensional curves of Fig. 3 show the effects of firing temperature, firing time and DWTR proportion on P adsorption capacity and a mass loss ratio. For P adsorption capacity (Fig. 3(a)), the increasing temperature has a negative effect especially when the temperature is over 800°C and the firing time is 60 min. Previous studies demonstrated that the P adsorption capacity of DWTR depends mainly on the content of amorphous Al [29], while the organic matter in DWTR also contributes to the P removal by DWTR [17]. At relatively low temperature, there were little major changes of amorphous Al in DWTR [17]. As a result, little changes of the P adsorption capacity within 600°C–700°C were found at all DWTR percentage (Fig. 3(b)). However, change of amorphous Al in the pellet at higher temperature occurred due to the bloating and crystallization caused by the oxidation and volatilization of inorganic substances [25]. As a result, there is a significant reduction of P adsorption at high firing temperature. In contrast, the P adsorption ability was improved with a higher DWTR proportion (Fig. 3(b)) because of the higher adsorption capacity of DWTR compared with kaolin clay. The firing time played a supportive role in adsorption capacity when the firing temperature is high (Fig. 3(a)) or the content of DWTR is low (Fig. 3(c)). However, longer firing time contributes to a better adsorption capacity at low firing temperature with a high DWTR content. The reason for these variations could be much more complex, including changes in material structure, surface characteristics, and needs to be further studied.

A significant decrease in mass loss ratio is observed when the kaolin clay was added (Fig. 3(d)), which indicated that the strength of DWTR could be increased by thermal treatment with kaolin clay. Though there was little strength fluctuation of samples with 15%–20% DWTR addition in DWTR clay mixture [30], this study focuses on the 30%–70% DWTR addition. A dramatic increase in mass loss ratio was observed when the DWTR ratio is over 60%. On the contrary, the mass loss ratio decreases with an increasing temperature (Fig. 3(e)). When the temperature is higher than 900°C, the mass loss ratio is close to 0 due to the neck growth by different types of crystallization linked to Al, Si and Ca [31].

Table 2
Experimental design and results

Run	Factor 1	Factor 2	Factor 3	Response 1	Response 2
	Time (min)	DWTR proportion (%)	Temperature (°C)	P adsorption capacity[a] (mg/g)	Mass loss ratio (%)
1	60	50	600	6.4845 ± 0.2308	0.2000 ± 0.0100
2	60	70	800	10.8496 ± 0.3711	0.1601 ± 0.0033
3	35	50	800	5.0639 ± 0.1519	0.0910 ± 0.0036
4	10	50	600	6.0289 ± 0.1809	0.1604 ± 0.0021
5	60	50	1,000	3.1027 ± 0.0931	0.0118 ± 0.0006
6	10	30	800	5.4277 ± 0.1628	0.0027 ± 0.0001
7	10	50	1,000	4.1543 ± 0.1246	0.0143 ± 0.0007
8	35	50	800	4.7562 ± 0.1427	0.0722 ± 0.0036
9	35	50	800	4.9084 ± 0.1473	0.0291 ± 0.0015
10	60	30	800	4.9496 ± 0.1485	0.0093 ± 0.0005
11	35	50	800	5.5486 ± 0.1665	0.0395 ± 0.0020
12	35	30	1,000	1.9612 ± 0.0588	0.0008 ± 0.0001
13	35	70	1,000	5.5365 ± 0.1661	0.0091 ± 0.0005
14	10	70	800	7.4730 ± 0.2242	0.2827 ± 0.0091
15	35	30	600	5.5097 ± 0.1653	0.0919 ± 0.0046
16	35	50	800	5.8358 ± 0.1751	0.0590 ± 0.0030
17	35	60	600	8.4268 ± 0.2528	0.2068 ± 0.0093

[a]Adsorption isotherm tests have been done under a solid/liquid ratio of 1 g/100 mL with an initial P concentration ranging from 0 to 3,500 mg/L for 96 h. The temperature is 25°C and the pH has not been controlled.

Table 3
ANOVA analysis for RSM

Source	Sum of squares	Degree of freedom	Mean square	F value	Prob > F
Adsorption capacity					
Model	59.6600	9	6.6300	11.9336	0.0018
Residual	3.8900	7	0.5600		
Lack of fit	3.1900	4	0.8000	3.4100	0.1704
Pure error	0.7000	3	0.2300		
R-squared	0.9388				
Mass loss ratio					
Model	0.1737	9	0.0193	4.3183	0.0334
Residual	0.0313	7	0.0045		
Lack of fit	0.0221	4	0.0055	1.7993	0.3285
Pure error	0.0092	3	0.0031		
R-squared	0.8474				

Although longer firing time contributes to a better strength (less mass loss ratio) at high firing temperature and reduces the strength (high mass loss ratio) at low firing temperature (Fig. 3(f)), this factor is not such significant as temperature and DWTR proportion.

Previous studies of DWTR thermal treatment have focused solely on one aspect, namely improving the adsorption capacity or comprehensive strength of the DWTR [17–19,24]. It is the first time in this study, trying to explore a balance point in thermal treatment for P adsorption capacity and comprehensive strength, as the pellets should have the potential to adsorb P with a relative good comprehensive strength when applied as adsorbent/substrate in wastewater treatment facilities such as constructed wetland, AFI or biofilter [32,33]. However, a higher DWTR ratio contributes better P adsorption ability but greater mass loss ratio. In terms of the effects of firing temperature, the situation is similar. Thus, it is important to find the balance point for adsorption ability and mass loss ratio with a suitable firing temperature, firing time and DWTR proportion. The optimized conditions predicted by RSM were firing at 650°C for 60 min with 60% DWTR and 40% kaolin clay. As such, the predicted P adsorption capacity and mass loss ratio were 10.3 mg P/g pellet and 0.2%, respectively.

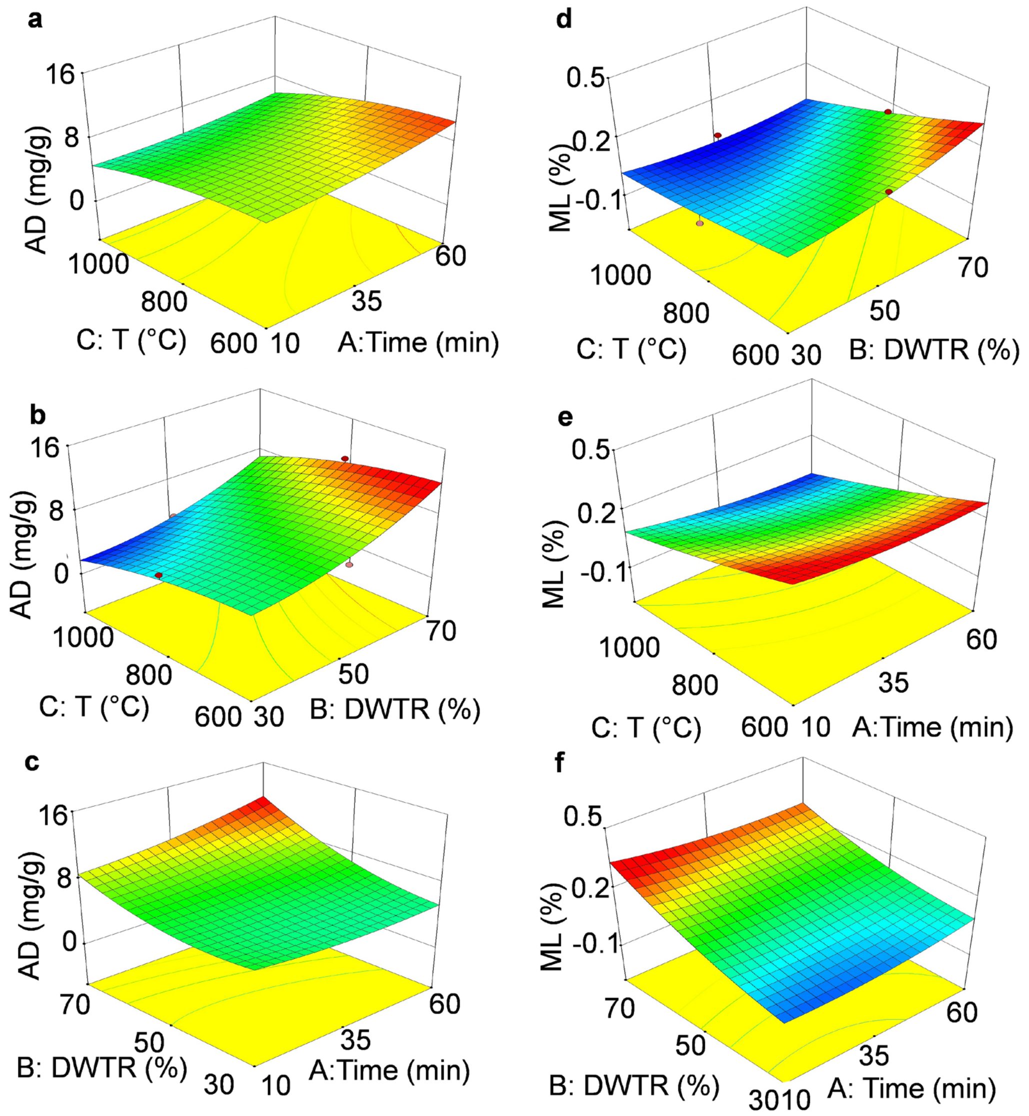

Fig. 3. Response surface curves for P adsorption capacity (AD) and mass loss ratio (ML) showing interaction between (A) time, min, (B) DWTR proportion, %, (C) temperature, °C.

3.2. Validation of the P adsorption capacity and mass loss ratio

In order to verify the optimal results, an experiment was performed under the predicted optimal conditions. The raw materials of DWTR (60%) and kaolin clay (40%) were first mixed. The same procedure was followed to make the pellets before they were subjected to firing in the furnace at 650°C for 60 min. Thereafter, the P adsorption test was conducted and modified by the Langmuir isotherm (Fig. 4). The Langmuir isotherm is a theoretical equation to describe the chemical or physical interaction postulated to occur between the solute and the available sites on the sorbent surface [34]. The linear form of Langmuir equation is given as:

$$\frac{c_e}{q_e} = \frac{c_e}{q_{\max}} + \frac{1}{q_{\max} k_L} \tag{4}$$

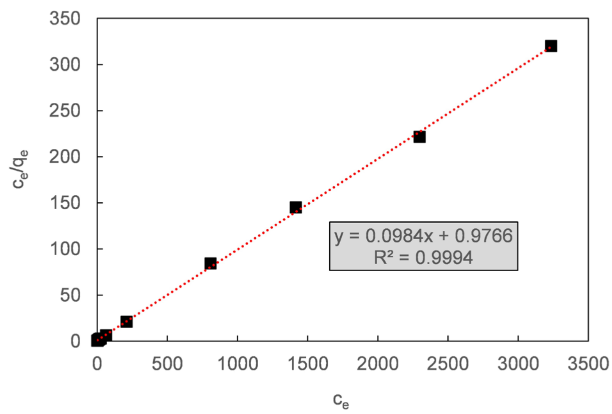

Fig. 4. P adsorption isotherm of pellet-type adsorbent.

where k_L (L/g) is Langmuir constant, c_e (mg/L) is the equilibrium concentration, q_e (mg/g) is the solute amount adsorbed at equilibrium and q_{max} (mg/g) represents the maximum adsorption capacity of complete monolayer coverage. A plot of c_e/q_e vs. c_e is given as a straight line with the slope $1/q_{max}$ and the intercept $1/k_Lq_{max}$. The R^2 value of Langmuir isotherm plots is 0.9994, indicating that this model can be used to describe the adsorption isotherm. The maximum adsorption of the pellet is 10.2 mg P/g pellet, while the result of the mass loss ratio experiment is 0.2%. These results closely agreed with the results obtained from RSM and hence validated the optimizing findings. Compared with other man-made materials in treatment wetland, the P adsorption capacity of this pellet-type adsorbent is relative good, as the P adsorption capacity of most materials ranges from 0.052 to 9.87 mg P/g material, only a lab-made lightweight aggregate has a higher P adsorption capacity with 12 mg P/g [32]. On the other hand, it is also a showcase that the comprehensive strength of DWTR could be increased by thermal treatment with kaolin clay.

4. Prospects for application of pellet-type adsorbent

In this study, a novel strategy for P immobilization with the DWTR-based adsorbent is proposed by making the DWTR as a pellet-type material for a better use of DWTR for P-rich wastewater treatment. It is expected and believed that the resultant DWTR pellets will extend the scope of the wide range of DWTR reuse and application in civil and environmental engineering. For instance, the pellet-type adsorbent can be used as material to build buffer zones besides agriculture areas to immobilize P. These shaped pellets can also be used as the substrate in constructed wetlands to relieve the clogging problem, which is commonly faced in constructed wetland practice in water pollution control. Moreover, it is believed that the concern of heavy metals leaching could be overcome through such utilization techniques, as heavy metals get intact when DWTR is sintered at higher temperature [1].

More significantly, it has been proposed in our group to use the DWTR pellet adsorbent as a matrix in AFI (Fig. 5) to enhance its treatment efficiency. Currently, the most striking drawback of the AFI lies in the fact that the treatment process of AFI depends solely on the vegetation (mainly roots) as it has no substrate or matrix, which has been well recognized to play a key role in constructed wetland-based technologies for wastewater treatment. As simulated in Fig. 5, it is reasonable to believe that the introduction of DWTR pellet adsorbent in AFI should have a better P immobilization from the water body while more surfaces from the pellets were provided for microbes to purify the water by biological activities. Compare with the direct application of DWTR powder into lake and reservoir [35], the proposed approach owns advantages not only on water quality improvement but also on the possibility to reuse the adsorbed P when the pellets were subjected for routine replacement after the long period use in AFI.

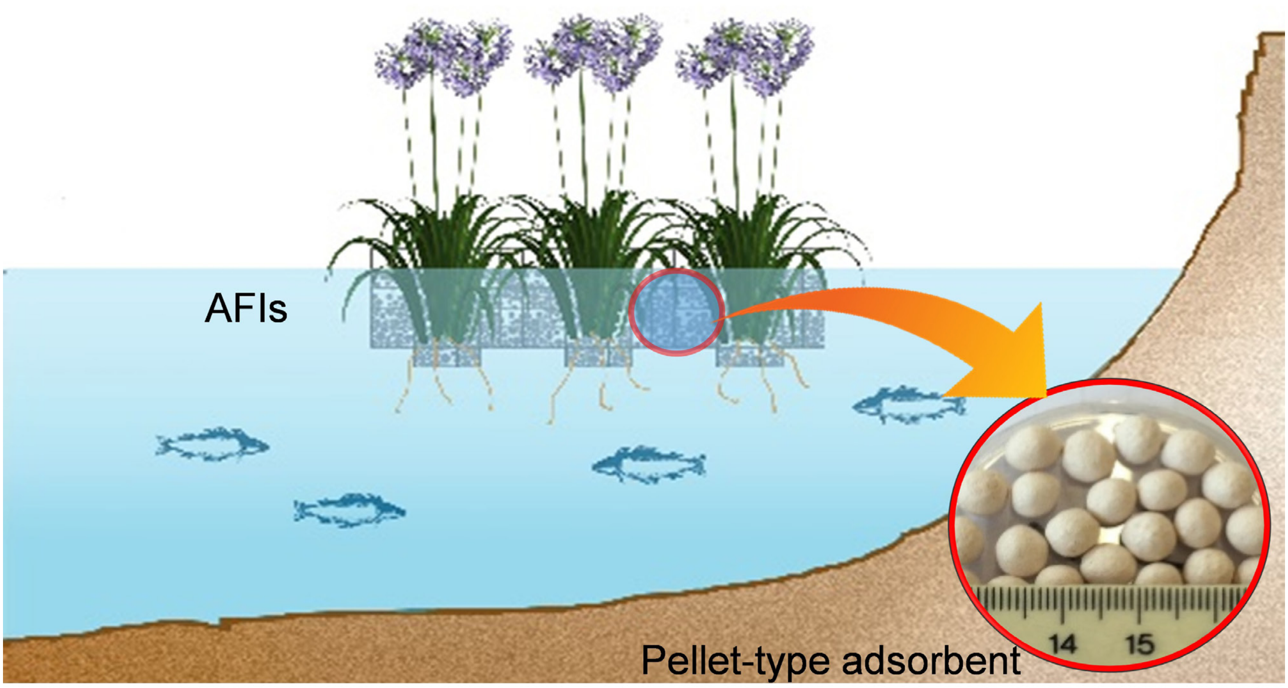

Fig. 5. Prospective application of pellet-type adsorbent as substrate in AFIs.

5. Conclusion

The DWTR-based pellet adsorbent with regular physical appearance and good strength can be achieved by thermal treatment with kaolin clay addition. However, increasing temperature contributes to greater strength but smaller P adsorption capacity. Similarly, Kaolin clay addition results in an improvement of strength but a decrease of P adsorption capacity. Under this situation, RSM was utilized to determine the balance point of the three factors of firing temperature, firing time and DWTR proportion for the best P adsorption capacity and a relatively good strength of pellets. The optimum condition of DWTR-clay pellet preparation process is at 650°C for 60 min with 60% DWTR proportion, at which the adsorption capacity of 10.3 mg P/g and 0.2% mass loss ratio can be achieved. The replicate experiments at optimal conditions yielded a P adsorption capacity of 10.2 mg P/g and a mass loss ratio of 0.2%, which shows the high agreement of predicted level using the established polynomial equation. By using RSM, two multi-variable polynomial equations have been developed to describe the preparation process of DWTR-clay pellets regarding the responses of adsorption capacity and mass loss ratio, respectively. These equations could be used as references for other application of DWTR-clay pellets making.

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