

## Chemical and Surface Properties of Activated Carbon from Banana Peel by Dry Chemical Activation

Supachai Wanprakhon<sup>a, b, \*</sup>, Phurin Pattaraphutanon<sup>b</sup>,  
Purana Borvornsudhasin<sup>b</sup>, Nannapas Choocherd<sup>b</sup>

<sup>a</sup> Division of Chemistry, School of Science, University of Phayao, Phayao, 56000 Thailand

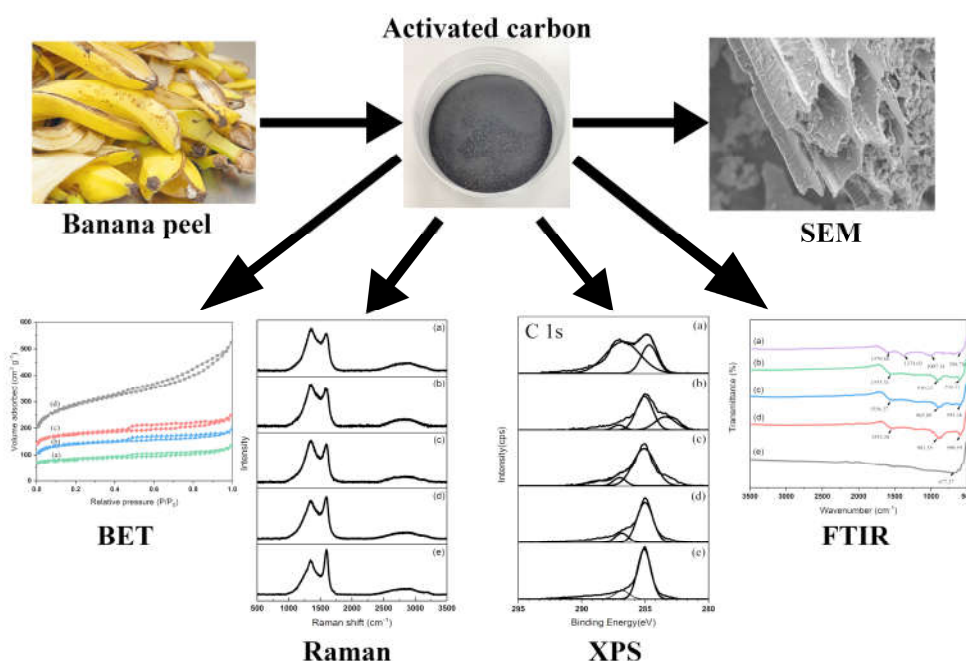
<sup>b</sup> Demonstration School University of Phayao, Phayao, 56000 Thailand

\*Corresponding Author: supachaixxxwanprakhon@gmail.com

Received 25 January 2021; Revised 2 February 2021; Accepted 20 August 2021; Available online: 1 September 2021

### Abstract

Throughout this research, chemical and surface properties of activated carbon were examined by taking into consideration dry chemical activation combined with  $\text{ZnCl}_2$  at the weight ratios between charcoal and  $\text{ZnCl}_2$  of 1:1, 1:2, 1:3 and 1:4 by activation temperature of 500 °C, 600 °C and 700 °C. The prepared activated carbon was then characterized using iodine number test method, Brunauer-Emmett-Teller (BET) surface area, Fourier transform infrared spectroscopy (FTIR), Raman spectroscopy, X-ray photoelectron spectroscopy (XPS), Scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS). The results showed that the optimum ratio and temperature was found at 1:3 and 700 °C, respectively. Which indicated improved efficiency of activated carbon prepared from banana peels.



**Keywords:** Activated carbon; Banana peel;  $\text{ZnCl}_2$ ; Dry chemical activation

## Introduction

Activated carbon is a product with porosity and elevated specific surface area which is procured from organic raw materials with carbon as the main component through physical activation or chemical activation process, which used widely in various applications beyond adsorption to treat various industrial effluents, especially activated carbon is one of the best means for dealing with water and air pollution issues. But the problem is that high-quality activated carbon is quite expensive. Therefore, the preparation of high quality activated carbon from waste products at low cost is a challenge for the present study [1 – 3].

The materials as precursors can be obtained by-product or waste of the agricultural and food industries with various activation agents and benefits such as fruit peel and cow dung waste activated by  $\text{H}_3\text{PO}_4$  for modified electrode fabrication, solid pineapple waste activated by  $\text{ZnCl}_2$  for removal of dye from waste water, macadamia nutshells activated by  $\text{H}_2\text{SO}_4$  and  $\text{K}_2\text{CO}_3$  for methylene blue removal in water, eggshell activated by  $\text{NaOH}$  and  $\text{H}_3\text{PO}_4$  for degradation of methylene blue via photocatalysis and bambusa vulgaris striata activated by  $\text{KOH}$  as an adsorbent in many industrial applications [4 – 8].

Banana peels are waste that are found in a food product. They were found that the percent yields of charcoal and wood vinegar from pyrolysis of banana peel are 57% and 7.53%, respectively [9]. In addition, they also contain 14% lignin, 14.8% hemicellulose and 13.2% cellulose which can be used as a raw material for the production of activated carbon [10 – 13].

The chemical activation process is an established method for obtaining a high surface area of activated carbons.  $\text{ZnCl}_2$  is one of the most chemical activating agents employed for organic materials, since it gave rise to activated carbons with a much wider porosity and more micropore structure depending on the activation temperature and impregnation ratio [14 – 24]. The activating agents react by dehydration of samples and interrupt the formation of volatiles and tar, thus increasing the activated carbon yield during the activation process [25].

Nevertheless, a conventional activating in which soak sample into a solution of the activated agent and dried by heating is called the wet chemical activation, which has many steps and time-consuming. To reduce steps and time, it is easier to directly mix the activation agents in a solid-state with a dry chemical activation [25]. This process was performed to investigate chemical and surface properties of activated carbon.

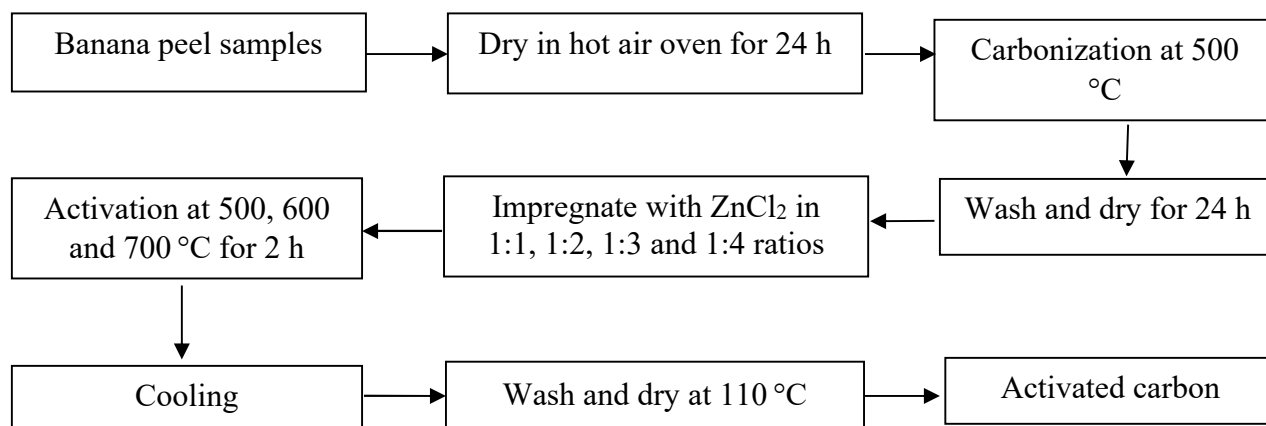
During this research, activated carbons were readied from banana peels by carrying out dry chemical activations with  $\text{ZnCl}_2$ . The iodine number, surface area, pore volume and SEM morphology gathered from the activated carbon were determined to describe the effect of activation temperature and the mass ratio of  $\text{ZnCl}_2$  on chemical and surface properties.

## Materials and Methods

### *Sample Preparation*

The dry banana peel samples were utilized for charcoal along with activated carbon precursors. Charcoal was specifically prepared by a carbonized temperature of 500 °C for 2 h, operating under a closed system formed by a porcelain crucible. Once this step was successful, the formed charcoal was then cooled to room temperature [9]. Furthermore, the charcoal was then activated by using a dry chemical method at the weight ratios between charcoal and  $\text{ZnCl}_2$  of 1:1, 1:2, 1:3

and 1:4. Consequently, the mixed samples were heated at preset activation temperatures ranging from 500, 600 and 700 °C. These specific temperatures were maintained constantly for a period of 2 h before cooling. Upon cooling down at room temperature, the activated carbon was then rinsed with water until the pH level of the carbon become neutral. The washed samples were then dried at 110 °C and stored for analysis (Fig. 1).



**Fig. 1** Steps for preparation of activated carbon from banana peel samples.

### *Sample Characterization*

The iodine number was gauged following the ASTM D4607-94 procedure. In this method, a proportion of charcoal and activated carbon was augmented to 10 mL of 5% HCl solution and then boiled before eventually being cooled. Next, 100 mL of 0.10 N iodine solution was added, shaken for 30 s and filtered. The 50 mL of filtrate was titrated with 0.10 N sodium thiosulphate solution with starch as an indicator. The iodine number is determined by the following eq. (1);

$$\frac{X}{M} = \frac{\left\{ (N_1 \times 126.93 \times V_1) - \left[ \left( \frac{V_1 + V_{HCL}}{V_F} \right) \times (N_2 \times 126.93 \times V_1) \right] \right\}}{M_C} \quad (1)$$

Where  $X/M$  is the iodine number value,  $N_1$  is the normality for iodine solutions,  $V_1$  relates to the added volume of iodine solution,  $V_{HCL}$  is the added volume of 5% HCl solution,  $V_F$  is the filtrate volume used in titration,  $N_2$  represents the normality of the sodium thiosulfate solution,  $V_2$  is the consumed volume of sodium thiosulfate solution and  $M_C$  is the mass of activated carbon.

The Brunauer-Emmett-Teller (BET) surface area was calculated by analyzing the surface area and pore size, the quantity of the gained sample was placed into a glass tube and then inserted into the instrument, thereby allowing the surface area to be obtained automatically. The functional groups of the activated carbon were analyzed using a Fourier transform infrared spectrophotometer (FTIR) the spectra were recorded in the region of 0 – 4,000  $\text{cm}^{-1}$ . Similarly, the Raman spectra were analyzed in the same range using a single monochromatic source for spectral light. X-ray photoelectron spectroscopy (XPS) was measured to determine the binding states of the elements in charcoal and activated carbon. Scanning electron microscopy (SEM) and energy-

dispersive X-ray spectroscopy (EDS) were used to visualize the surface morphology and elemental composition of the charcoal and activated carbon. The samples were coated with gold by a gold sputtering device for a clear vision of the surface morphology of the samples.

## Results and Discussion

The iodine number pertained from the activated carbon was calculated at a varied ratio of charcoal:  $\text{ZnCl}_2$  and activation temperatures, as shown in Table 1, it was found that the iodine value obtained from dry chemical activation increased in proportion to the mass ratio of the charcoal powder to the activated reagent from 1:1 ratio to 1:3 ratio, then has a reduced value at 1:4 ratio in the temperature range from 500 – 700 °C, possibly due to an excess of  $\text{ZnCl}_2$ , and it is possible that in the activation process  $\text{ZnCl}_2$  reacts with the ash contained in the charcoal [26]. This reduces the number of  $\text{ZnCl}_2$  that reacts with charcoal. Also, the highest value of iodine number at 630  $\text{mg g}^{-1}$  under activation temperature of 700 °C of ratio 1:3 was found and the optimum iodine number at each temperature is 1:3 ratio of charcoal:  $\text{ZnCl}_2$ . However, it was found that the iodine number was significantly lower when compared to commercial activated carbon.

**Table 1** Iodine number of activated carbon with different ratio of  $\text{ZnCl}_2$  and commercial grade.

Activation temperature (°C)	Charcoal : $\text{ZnCl}_2$	Iodine number ( $\text{mg g}^{-1}$ )
500	1:1	353
	1:2	480
	1:3	561
	1:4	520
600	1:1	373
	1:2	570
	1:3	588
	1:4	586
700	1:1	454
	1:2	603
	1:3	630
	1:4	592
Commercial grade	-	1098

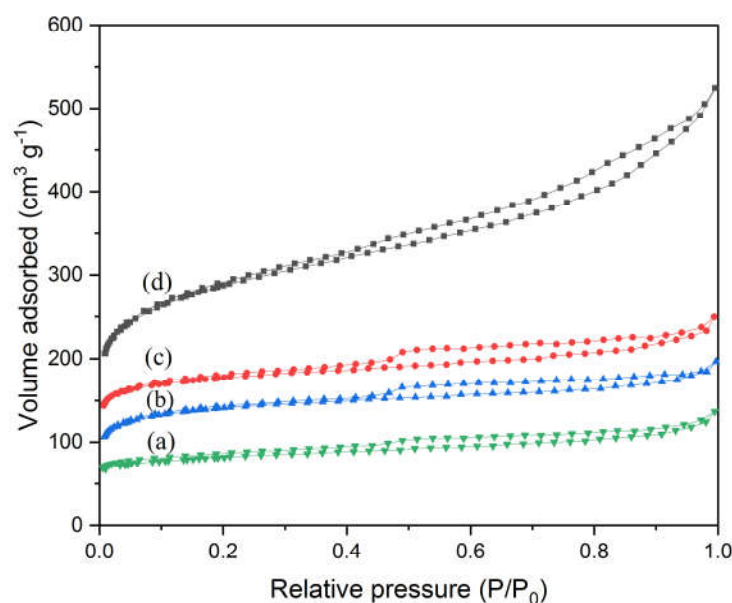
**Table 2** Iodine number of activated carbon with different ratio of  $\text{ZnCl}_2$  and commercial grade.

Activation temperature (°C)	Surface area ( $\text{m}^2 \text{g}^{-1}$ )	Pore volume ( $\text{cm}^3 \text{g}^{-1}$ )
500	311.18	0.22
600	542.43	0.32
700	625.82	0.37
Commercial grade	1034.05	0.81

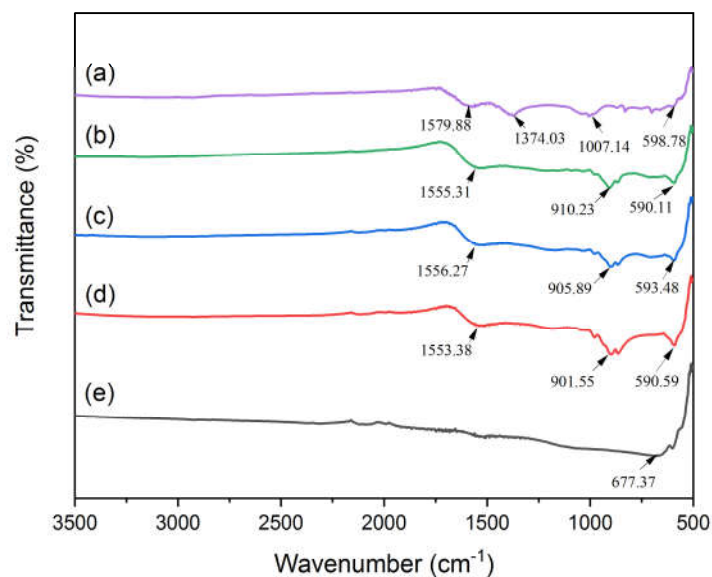
From Table 2, we can see that the surface area and pore volume obtained from activated carbon that was prepared under an optimum ratio of charcoal:  $\text{ZnCl}_2$  and activation temperature were 625.82  $\text{m}^2 \text{g}^{-1}$  and 0.37  $\text{cm}^3 \text{g}^{-1}$ , respectively. Figures show that by increasing activation

temperature slightly, the surface area and pore volume of activated carbon which is due to the evaporation mechanism of  $\text{ZnCl}_2$  into zinc and chlorine, which can further create and enlarge the pores [27]. However, these results were still less than that of commercial activated carbon with high surface area and pore volume. Similarly, the adsorption isotherm in Fig. 2 show that the sequence of adsorption capacity is commercial grade (Fig. 2 (d)) > activated carbons of ratio 1:3 at 700 °C (Fig. 2 (c)) > 600 °C (Fig. 2 (b)) > 500 °C (Fig. 2 (a)). This result tends to be the same surface area and pore volume as shown in Table 2.

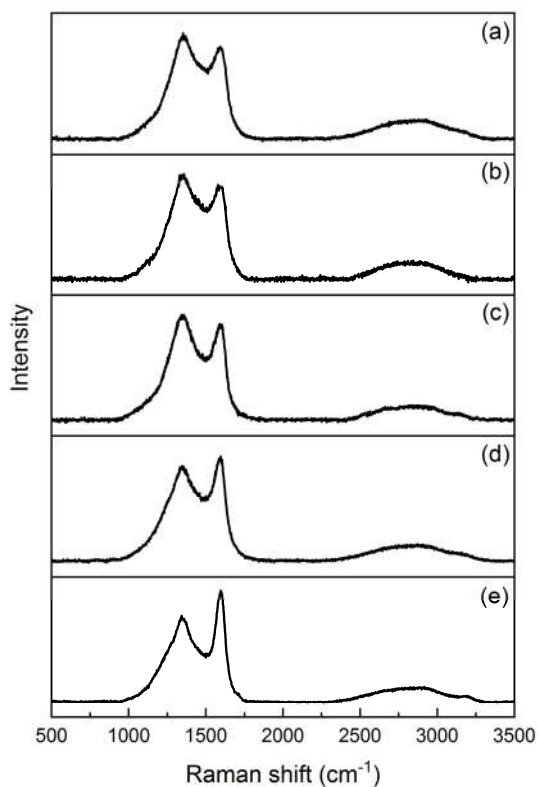
Figure 3 (a) presented the FTIR spectra of charcoal before activation process shows the absorption peak at 1579.88, 1374.03, 1007.14, and 598.78  $\text{cm}^{-1}$  of C=C, C-O, C-O-C and C-C groups respectively. Whereas, for the spectra of activated carbon after activation process, the position of some peaks being changed (Fig. 3 (b), (c) and (d)). Peak around 1374  $\text{cm}^{-1}$  became shallower and peak around 1000  $\text{cm}^{-1}$  was slightly shifted to around 900  $\text{cm}^{-1}$  which suggested the decrease of cellulose and lignin content. In addition, when compared with commercial activated carbon the peak was found at 677.37  $\text{cm}^{-1}$  meaning that the increasing of C-C group.



**Fig. 2** Nitrogen adsorption – desorption isotherm of activated carbon with  $\text{ZnCl}_2$  activation of ratio 1:3 and activation temperature at (a) 500 °C, (b) 600 °C, (c) 700 °C and (d) commercial grade.

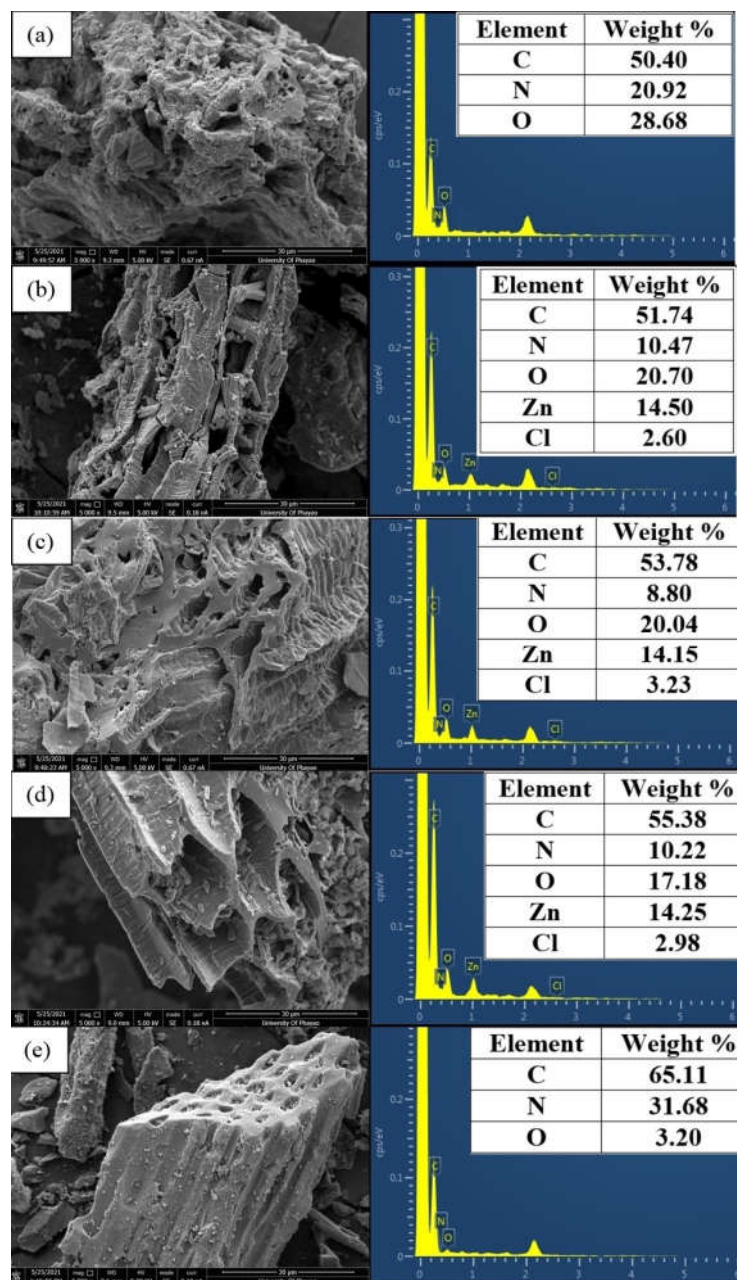


**Fig. 3** FTIR spectra of (a) charcoal, activated carbon with  $\text{ZnCl}_2$  activation of ratio 1:3 and activation temperature at (b) 500 °C, (c) 600 °C, (d) 700 °C and (e) commercial grade.



**Fig. 4** Raman spectra of (a) charcoal, activated carbon with  $\text{ZnCl}_2$  activation of ratio 1:3 and activation temperature at (b) 500 °C, (c) 600 °C, (d) 700 °C and (e) commercial grade.

Figure 4 shows the Raman spectra for the banana peel charcoal and activated carbons. Two pronounced bands can be seen in all spectra at around 1350 and 1600  $\text{cm}^{-1}$ , these may be attributed to the D and G bands respectively. The D band is referred to disordered carbon structure, whereas G band is referred to graphitic structure or whiskers like carbon, which is more ordered, symmetrical and crystalline, which can be seen that the G band peak was higher than D band peak of activated carbon at a ratio of 1:3 at 700 °C (Fig. 4(d)), indicating that more crystalline structure [28]. This is especially evident in the case of commercial activated carbon as showed in Fig. 4 (e).



**Fig. 6** SEM – EDS of (a) charcoal, activated carbon with  $\text{ZnCl}_2$  activation of ratio 1:3 and activation temperature at (b) 500 °C, (c) 600 °C, (d) 700 °C and (e) commercial grade.

Figure 6 shows the surface morphology of charcoal and activated carbon that were visualized by the SEM at 5000X magnification of charcoal and activated carbon with  $\text{ZnCl}_2$  activation of ratio 1:3 at the activation temperature from 500 – 700 °C. From Fig. 6(a), (b) and (c), were seen the less porous structures of activated carbon. Whereas, Fig. 6(d), a porous structure was apparent due to the volatile matters can be more removed at the activation temperature of 700 °C [30]. This results show the opportunity to further improve surface area of porous activated carbon through the optimization of  $\text{ZnCl}_2$  mass ratio and activation temperature. Whereas the surface morphology of commercial activated carbon has a relatively smaller but uniform pore size. Furthermore, EDS analysis results indicated that activated carbon with  $\text{ZnCl}_2$  activation of ratio of 1:3, there was an increase in the carbon composition from 51.74, 53.78 and 55.38 at 500, 600 and 700 °C, respectively. Increased composition of carbon affects the adsorption capacity of activated carbon. However, there are still about 14% of zinc on the surface of the activated carbon we can assume that evaporation of  $\text{ZnCl}_2$  and homogeneously covers the surface of the activated carbon.

## Conclusion

The activated carbon produced from banana peels were prepared by  $\text{ZnCl}_2$  activation. When using 1:3 ratio of charcoal and  $\text{ZnCl}_2$  by activation temperature at 700 °C indicated the optimum iodine number, surface area, pore volume, crystalline structure of carbon and surface morphology were found. However, these results are still lower when compared to the commercial activated carbon. In addition, dry chemical activation method was also found to contain high amounts of zinc caused by the evaporation mechanism and it is homogeneous with the activated carbon surface which requires further improvement.

## Acknowledgement

This research was supported by the School of Science, University of Phayao and the Demonstration School University of Phayao. The authors also thank the anonymous reviewers for their useful comments to improve the quality of the paper.

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