

## **Adsorption and Desorption Performance of Stem Torch Ginger for the Release of Niacinamide from Aqueous Solution**

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### **Abstract**

Base treated stem torch ginger (BTSTG) was used as natural adsorbent for niacinamide (vitamin B3), a whitening agent in cosmetic products. Batch adsorption and desorption experiments were conducted. The equilibrium time of BTSTG for the adsorption of niacinamide at 30 °C was 150 minutes. The adsorption behavior fitted well with the Freundlich isotherm. A pseudo first order equation described the adsorption better than a pseudo second order. Thermodynamic parameters were evaluated: a positive value of  $\Delta H^\circ$  suggested that the adsorption process is endothermic and negative values of  $\Delta G^\circ$  indicated the spontaneous nature of the adsorption. The desorption results showed that BTSTG can release up to 39.7 mg/g of niacinamide. The results of this study confirm that stem torch ginger can act as biosorbent for niacinamide and could be applied as a novel scrub component in cosmetic products.

**Keywords:** Adsorption, Natural adsorbent, Stem torch ginger, niacinamide, Isotherm.

## INTRODUCTION

The scientific name of torch ginger is *Etilingera elatior* (Jack) R. M. Smith. *E. elatior* is used as a species of herb plant, especially in cuisines of Thailand and Indonesia. This plant is a species of herbaceous perennial herb plants believed to be indigenous of South Thailand, South East Asia and tropical regions of the world. This herb plant belongs to the family of Zingiberaceae such as ginger, torch ginger, galangal, turmeric, and curcumin etc. Torch ginger is one type of native plants found in Thailand, Indonesia, Vietnam, Malaysia and other countries in Southeast Asia. The inflorescence of torch ginger looks like torches. In the recent years, torch ginger has become also an ornamental plant as their flowers are colorful. The inflorescence of torch ginger contains important nutrition substances such as protein (12.6%), fat (18.2%) and fiber (17.6%) [1]. Torch ginger is widely consumed by indigenous communities as condiment, eaten raw or cooked as vegetable and used for food flavouring [2]. Leaves of torch ginger, mixed with other aromatic herbs, are used for bathing to remove body odor of post partum women [3]. Previous studies on the antioxidant property of leaves of *E. elatior* showed their antibacterial property [4], strong antioxidant activity [5] and good smell of its essential oil [6] as well as other pharmacological properties. Torch ginger has great potential to be developed into functional and other health products. Torch ginger is also used as an ingredient for local products such as soap, shampoo and perfume [7]. After extraction of the important reagents from the flowers of torch ginger, the torch ginger will become waste. Literature reviews from previous studies on the adsorption properties of plants of Zingiberaceae revealed that ginger can adsorb patent blue VF [8], malachite green [9] and crystal violet [10]. Turmeric can adsorb aluminium [11], and Galangal lead and zinc ions [12]. Moreover, plants of torch ginger are tall with long filaments. These plants can grow up to 5-6 m tall [6]. Torch ginger contains water insoluble fibers [13]. From the information above, it can be hypothesized that torch ginger as a type of Zingiberaceae has fibers especially in the stem part. After extraction of the essential reagents from the stem, the fibers remain as waste material. These waste materials from the ginger stem are numerous and readily available. Research studies previously have used a sodium hydroxide solution to neutralize the surface of the fiber plants such as in the fiber preparation of luffa [14], torch ginger [15] and rice husk [16]. The sodium hydroxide solution has the ability to eliminate the gum [14]. Based on these results, this research evaluates a base treatment of the surface of stem torch ginger and then studies the ability to adsorb and release niacinamide (vitamin B3). Vitamin B3 is one type of whitening agent which is widely used in cosmetic products because it is an effective skin lightening agent which works by inhibiting the transfer of melanosome from melanocytes to keratinocytes [17]. The stem torch ginger waste material should be useful as adsorbent material by nature. However at present, no research has been done about the behavior and amount of adsorption and desorption of niacinamide solution on fibers of stem torch ginger. For the reasons mentioned above, the researchers are interested in the preparation of adsorbent materials from stem of torch ginger (natural waste material) to adsorb and release niacinamide. This is a new way to add value to agricultural waste by using it as scrub material in cosmetic products.

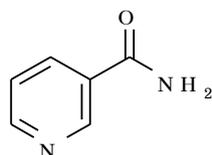
## EXPERIMENTAL SECTION

### Preparation of adsorbent materials

Adsorbent materials were prepared from fresh stem torch ginger by the method of Ahmad and Kumar [7], Syahrizul *et al.*, [15], Demir *et al.*, [18] and Ghali *et al.*, [19]. Stems of fresh torch ginger were collected from a local cuisine at Amphawa, Samut Songkhram Province, Thailand. They were used in the form of small pieces (0.149 mm) to adsorb and release niacinamide. These pieces of torch ginger were prepared by separation of the leaves and stems from torch ginger and washing the stems with distilled water to remove dirt. The stems were cut into small pieces to pass a sieve of size 100 mesh (0.149 mm) and then dried in an oven (Mettler, UM500) at 60 °C for 12 hours [7]. Stem torch ginger was then soaked into 0.25 M NaOH for 1 hour at room temperature [15], washed with distilled water to eliminate excess NaOH solution and dried again at 60 °C for 12 hours. The dried stem torch ginger was then grounded and sieved into 100 mesh (0.149 mm) particle size SUS304. The fibers were soaked into 1 M H<sub>2</sub>O<sub>2</sub> at 100 °C for 2 hours [18,19] and then rinsed thoroughly with distilled water until the pH value of the solution became equal to 7. After drying in an oven at 60 °C they were kept in a dry box for further experiments.

### Adsorbate

Niacinamide or nicotinamide is chemically pyridine-3-carboxamide, also known as amide of nicotinic acid (vitamin B<sub>3</sub>/niacin). The molecular formula of this organic compound is C<sub>6</sub>H<sub>6</sub>N<sub>2</sub>O with a molecular weight of 122.13 g/mol. It comes in the form of colorless crystals, soluble in distilled water. The molecular structure of niacinamide is illustrated in Figure 1.



**Figure1:** The structure of Niacinamide

### Study the zero point charge of stem torch ginger

0.5 gram portions of stem torch ginger, treated with base, were put into beakers, each beaker containing a buffer solution of pH 2—12 respectively. After soaking for 24 hours, each beaker content was filtered and the pH of the solutions were measured by a pH meter (Hanna, HI-9321).

### Batch adsorption studies

Exact amounts of base treated stem torch ginger of 0.50 g were added into different Erlenmeyer flasks. Niacinamide solutions at concentrations of 0.50, 1.00, 1.50, 2.00, 2.50 and 3.00 %w/v, were added to fill up to 50 mL of the flask. The solutions were shaken by a shaker (n-biotek, NB-205) at a speed of 120 rpm at 30 °C with different periods including 1, 5, 10, 20, 30, 40, 50, 60, 90, 120, 150, 180, 210, 240 and 270

minutes, respectively. The flasks were then removed from the shaker and the solutions filtered through filter paper to separate the stem torch ginger fibers from the solutions. The final concentration of niacinamide in the solutions was analyzed by a double beam UV-Visible Spectrophotometer (Shimadzu, UV-2401PC) at a wavelength of 262 nm, three times in repetition.

To study the effect of concentration and temperature, 0.50 g amounts of base treated stem torch ginger were added into different Erlenmeyer flasks. 50 mL of different concentrated solutions of niacinamide (0.50, 1.00, 1.50, 2.00, 2.50 and 3.00 %w/v) were added to each of the flasks. Each sample was kept in a shaker at different temperatures of 30, 40 and 50 °C for equilibrium time (150 minute). The adsorption capacity of niacinamide at equilibrium,  $q_e$  was calculated by equation (1).

$$q_e = \frac{(C_0 - C_e)V}{W} \dots\dots\dots(1)$$

- With  $q_e$  as adsorption capacity of niacinamide at equilibrium (mg/g)  
 $C_0$  is the initial concentration of the niacinamide solution (mg/L)  
 $C_e$  is the equilibrium concentration of the niacinamide solution (mg/L)  
 $V$  is the volume of the niacinamide solution (L)  
 $W$  is the weight of the base treated stem torch ginger (g)

### Thermodynamics study

0.50 g portions of base treated stem torch ginger were added into 250 mL Erlenmeyer flasks, containing different concentrated niacinamide solutions of 0.50, 1.00, 1.50, 2.00, 2.50 and 3.00 %w/v. Each flask was shaken by a shaker during equilibrium time (150 minutes) at a speed of 120 rpm, at various temperatures of 30, 40 and 50 °C. The concentration of niacinamide remaining in solution was determined by a UV-Visible Spectrophotometer at a wavelength of 262 nm with 1 cm matched quartz cells.

### Study of the surface characteristic of stem torch ginger

Untreated and treated stem torch ginger fibers were placed in 10 mm diameter aluminum stubs, gold coated to provide electrical conductivity. The surface characteristic of the stem torch ginger was analyzed by a Scanning Electron Microscope (SEM: JSM-7610F). The fibers were scanned under 1000x magnification.

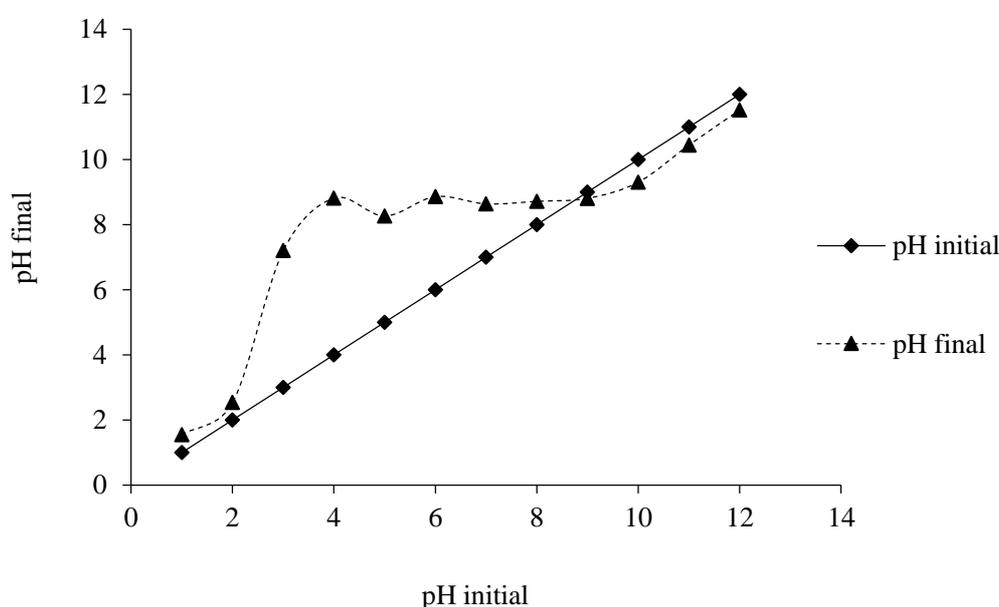
### Study the amount of desorption of niacinamide from stem torch ginger

0.50 gram amounts of base treated stem torch ginger, which had adsorbed niacinamide at a concentration of 0.5 %w/v, were added into Erlenmeyer flasks. 50 ml distilled water was added to each flask and shaken by a shaker for 1, 5, 10, 20, 30, 40, 50, 60, 90 and 120 minutes, respectively at 30 °C. The amount of niacinamide which was released from the adsorbent was determined by a UV-Visible Spectrophotometer at 262 nm.

## RESULTS AND DISCUSSION

### Point of zero charge of base treated stem torch ginger

From the research of Ahmad *et al.* [9], which studied the charge on the surface of ginger treated with sulfuric acid, it was found that ginger has a zero point charge equal to 6. In this research the charge on the surface of stem torch ginger treated with base is shown in Figure 2. The two curves intersect at a pH of 9 indicating that the base treated stem torch ginger has a zero point charge equal to 9. A pH less than 9 will result in a positive charge due to the amount of hydronium ions ( $H_3O^+$ ) on the surface of the stem torch ginger. Hydroxide ions ( $OH^-$ ) ions at a pH greater than 9 will cause negative charges [20]. In this experimental study the pH of the solutions was kept equal to 8 so that the surface of the stem torch ginger has a positive charge. In this way, the adsorbent can be attracted to the negative charges of niacinamide.

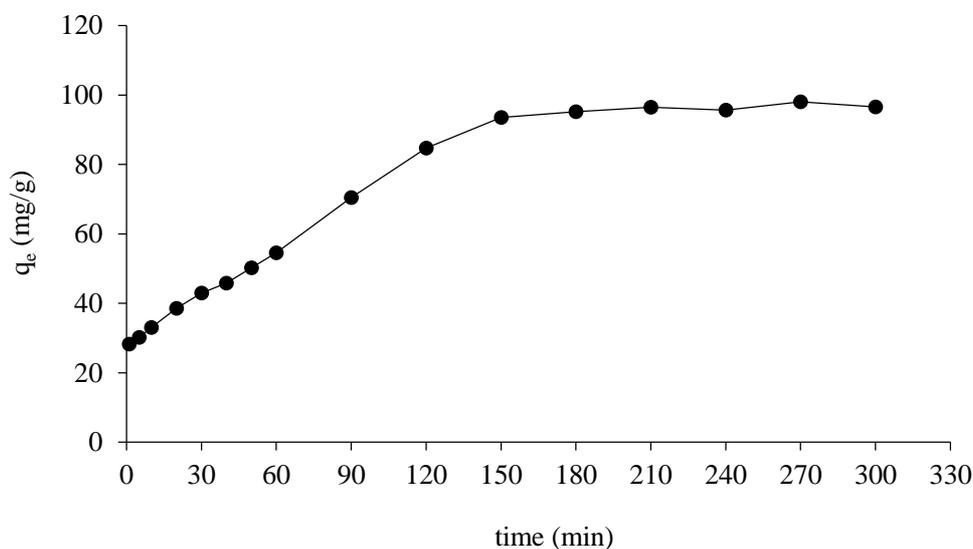


**Figure 2:** Point of zero charge of base treated stem torch ginger.

### Effect of contact time

The effect of contact time for the absorption of niacinamide is shown in Figure 3. In the beginning the graph is very steep and became parallel to the x-axis later at equilibrium time. That means a large amount of niacinamide was adsorbed rapidly during the initial 150 minutes of contact and then the absorption speed slowed down until equilibrium was achieved. The rapid initial adsorption could be attributed to the large amounts of adsorbent active sites available for adsorbate molecules [9].

In the early start time, the stem torch ginger can adsorb niacinamide quickly also because of a high surface area available for adsorption until the equilibrium time at 150 minutes. After 150 minutes, the active sites are just saturated with niacinamide molecules.

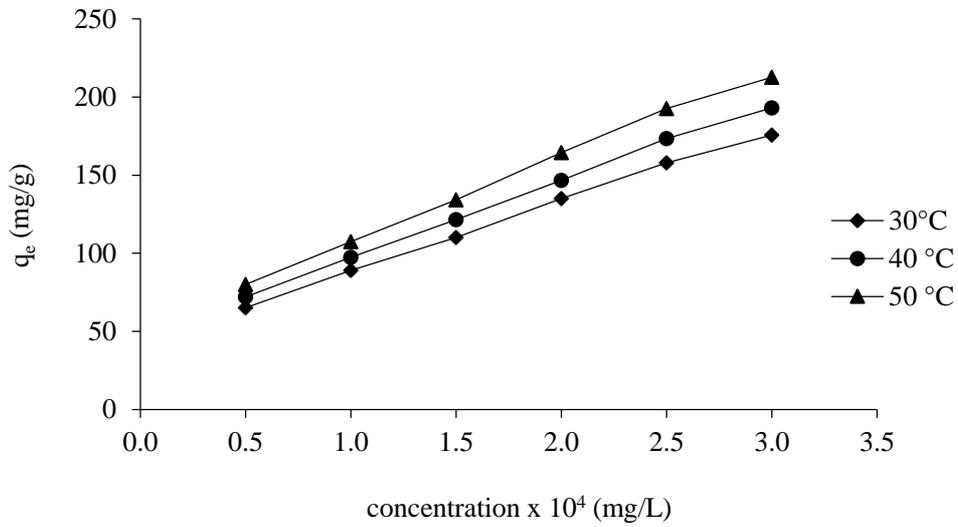


**Figure 3:** Effect of contact time for the adsorption of niacinamide on base treated stem torch ginger. (at a the solution pH 8, temperature 30 °C).

#### **Effect of concentration and temperature**

In the present study, the concentration of niacinamide solutions was varied from 0.50, 1.00, 1.50, 2.00, 2.50 and 3.00 %w/v. The effect of the concentration on the amount adsorbed is shown in Figure 4. According to Figure 4 the adsorption capacity of base treated stem torch ginger increases steadily when the niacinamide concentration is increased up to 3.0 %w/v indicating higher amounts of niacinamide molecules occupy the surface of the base treated stem torch ginger at higher concentrations [8].

In addition, Figure 4 shows the effect of temperature on the adsorption of base treated stem torch ginger at three different values (30, 40, 50 °C). When the temperature raises the base treated stem torch ginger can adsorb more niacinamide at all tested concentrations due to higher kinetic energy, so more adsorbate molecules can move onto the surface of the adsorbent [21]. This indicates that the adsorption of niacinamide on the surface of base treated stem torch ginger is an endothermic process [9].



**Figure 4:** Effect of concentration and temperature for the adsorption of niacinamide on base treated stem torch ginger at different temperatures. (at solution pH 8).

**Adsorption Isotherm**

Adsorption isotherms show the relationship between the amounts of niacinamide adsorbed per unit weight of base treated stem torch ginger at constant temperature. The absorption behavior of niacinamide on base treated stem torch ginger can be described by Langmuir and Freundlich isotherms as in equation (2) and (3), respectively.

$$\frac{1}{q_e} = \left( \frac{1}{q_{max} K_L C_e} \right) + \left( \frac{1}{q_{max}} \right) \dots\dots\dots(2)$$

$$\log q_e = \log K_F + \left( \frac{1}{n} \log C_e \right) \dots\dots\dots(3)$$

- When q<sub>e</sub> is the equilibrium loading of adsorbate per unit mass of adsorbent (in mg/g)
- C<sub>e</sub> is the equilibrium concentration of niacinamide solution (mg/L)
- q<sub>max</sub> is the maximum adsorbed niacinamide amount on monolayers (mg/g)
- K<sub>L</sub> is the Langmuir constant related to adsorption capacity (mg/L)
- 1/n is the adsorption constant (strength of adsorption) (mg/g)
- K<sub>F</sub> is the Freundlich constant related to rate of adsorption (mg/g)

The behavior of niacinamide adsorbed on the base treated stem torch ginger is summarized in Table 1. This table shows the values of the variables calculated from the Langmuir and Freundlich equation. The data show a good correlation coefficient (R<sup>2</sup>) for both the Langmuir and Freundlich equation. However, the adsorption

isotherm is important to describe how the adsorbate interacts with the adsorbent. The correlation coefficient of the Freundlich equation came closer to 1 than the Langmuir equation. That means the absorption behavior of niacinamide on base treated stem torch ginger corresponds slightly better with the Freundlich than the Langmuir equation. Therefore, the behavior of niacinamide molecules is best described as a multilayer absorption process on the surface of base treated stem torch ginger [21].

**Table 1:** Langmuir and Freundlich isotherms for adsorption of niacinamide on base treated stem torch ginger.

Temp. (°C)	Langmuir isotherm			Freundlich isotherm			
	q <sub>max</sub> (mg/g)	K <sub>L</sub> (mL/mg)	R <sup>2</sup>	K <sub>F</sub> (mg/g)	1/n (mL/mg)	n (mg/mL)	R <sup>2</sup>
30	204	0.101	0.952	2.21	1.84	0.544	0.988
40	222	0.106	0.947	1.64	1.86	0.537	0.986
50	243	0.109	0.942	2.21	1.87	0.534	0.985

Thermodynamic parameters, enthalpy change (enthalpy;  $\Delta H^\circ$ ), entropy change (entropy;  $\Delta S^\circ$ ) and Gibbs free energy change (Gibbs free energy;  $\Delta G^\circ$ ) can be calculated from Van't Hoff and Gibbs-Helmholtz equations (4) — (6) [8—10].

$$K_C = \frac{C_{ae}}{C_e} \quad \dots\dots\dots(4)$$

$$\Delta G^\circ = -RT \ln K_C \quad \dots\dots\dots(5)$$

$$\log K_C = \left( \frac{\Delta S^\circ}{2.303 R} \right) - \left( \frac{\Delta H^\circ}{2.303 RT} \right) \quad \dots\dots\dots(6)$$

When  $K_C$  is the equilibrium constant (dimensionless)

$C_{ae}$  is the amount adsorbed on base treated stem torch ginger at equilibrium (mg/100mL)

$C_e$  is the equilibrium concentration (mg/100mL)

$T$  is the absolute solution temperature (K).

$R$  is the universal gas constant (8.314 J/mol·K)

$\Delta H^\circ$  is the enthalpy change (kJ/mol)

$\Delta S^\circ$  is the entropy change (J/mol·K)

$\Delta G^\circ$  is the Gibbs free energy change (kJ/mol)

A linear plot of  $\log K_C$  versus  $1/T$  shows the values of  $\Delta H^\circ$  in the slope and of  $\Delta S^\circ$  in the intercept. The results of thermodynamics calculations are shown in Table 2. The

$\Delta H^\circ$  variables are positive what means that the adsorption of niacinamide on base treated stem torch ginger is an endothermic reaction [22].  $\Delta S^\circ$  also has a positive value indicating that the adsorption process is random without a definite direction [22]. Negative values of  $\Delta G^\circ$  suggest that the adsorption process is a spontaneous [22].

**Table 2:** Value of thermodynamic variables for the adsorption of niacinamide on base treated stem torch ginger.

Temp. (°C)	$K_c$	$\Delta H^\circ$ (kJ/mol)	$\Delta S^\circ$ (J/mol·K)	$\Delta G^\circ$ (kJ/mol)
30	1.09	0.82	3.40	-0.212
40	1.10			-0.242
50	1.11			-0.278

**Kinetics studies**

The kinetics to study the rate of adsorption of niacinamide on base treated stem torch ginger is based on pseudo first order and pseudo second order equations according to equations (7)-(8), respectively.

$$\log(q_e - q_t) = \log q_e - \left(\frac{k_1 t}{2.303}\right) \dots\dots\dots(7)$$

$$\frac{t}{q_t} = \left(\frac{1}{k_2 q_e^2}\right) + \frac{t}{q_e} \dots\dots\dots(8)$$

- Where  $q_e$  is the adsorption capacity of niacinamide solution at equilibrium (mg/g)
- $q_t$  is the amount of adsorbed niacinamide at each time (mg/g)
- $k_1$  is the rate constant of pseudo first order reaction (1/min)
- $k_2$  is the rate constant of pseudo second order reaction (mg/g·min)

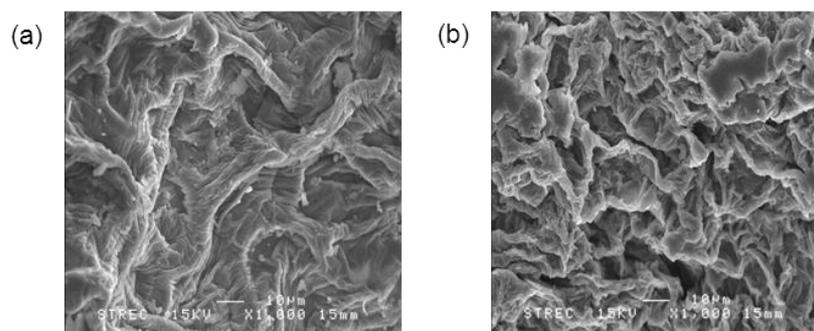
The kinetic experiment results (Table 3) showed that the correlation coefficient ( $R^2$ ) of a pseudo first order reaction is closer to 1 than  $R^2$  of a pseudo second order reaction. The adsorption of niacinamide on base treated stem torch ginger corresponded better to pseudo first order than pseudo second order. Comparing the amount of adsorption from the experiment calculated by pseudo first order and pseudo second order also shows that the pseudo first order equation fits better than the pseudo second order.

**Table 3:** Kinetic parameters for the niacinamide adsorption on base treated stem torch ginger.

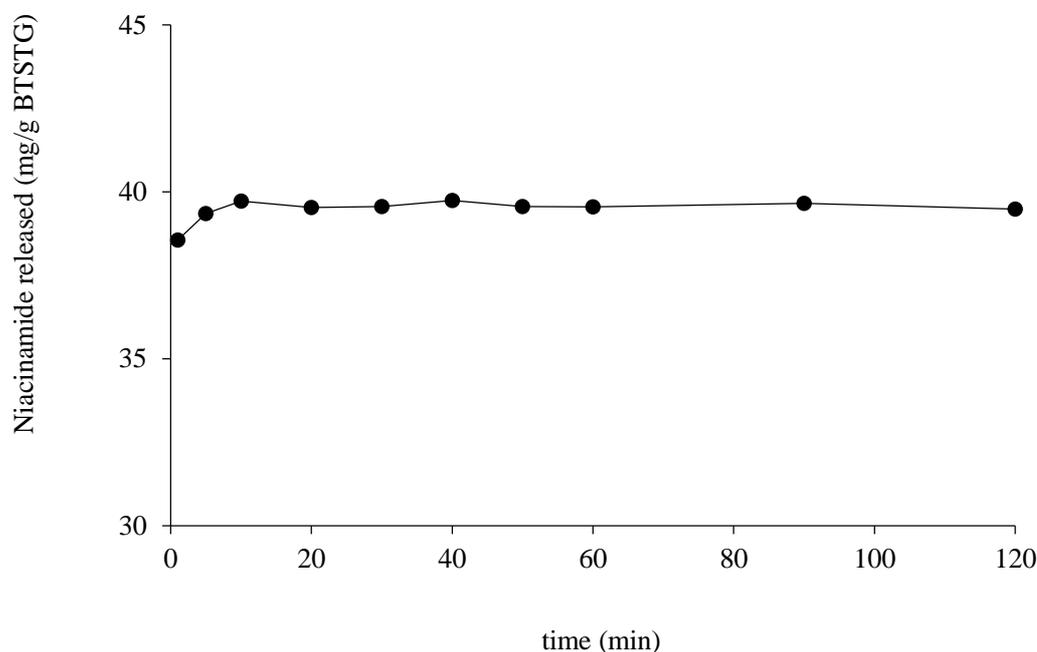
C <sub>0</sub> (% w/v)	q <sub>e</sub> (exp) (mg/g)	pseudo first order equation			pseudo second order equation		
		q <sub>e</sub> (cal) (mg/g)	k <sub>1</sub> (1/min)	R <sup>2</sup>	q <sub>e</sub> (cal) (mg/g)	k <sub>2</sub> (mg/g·min)	R <sup>2</sup>
0.5	64.8	56.6	0.0094	0.965	50.5	0.00073	0.874
1.0	95.2	77.6	0.0143	0.922	86.2	0.00055	0.894
2.0	135.9	111.6	0.0286	0.929	140.8	0.00050	0.957

**Surface morphology of the stem torch ginger**

The surface morphology of adsorbent material from stem torch ginger was characterized by scanning electron microscopy (SEM). SEM images clearly showed the roughness of the surface of stem torch ginger as in Figure 5 (a) compared to the characteristic surface of adsorbent material from base treated stem torch ginger as higher irregular (more roughness) as shown in Figure 5 (b). The base treatment increased the roughness of the surface of the fibers [23].

**Figure 5:** SEM images of the stem torch ginger waste material (a) before treated and (b) after treated with base at magnification of 1000x.**Desorption of niacinamide from adsorbent**

To study the release of niacinamide in the presence of water as a solvent, different time intervals (1, 5, 10, 20, 30, 40, 50, 60, 90 and 120 minutes) at constant temperature of 30 °C were chosen. It was found that the stem torch ginger treated with base can release niacinamide in an amount equal to 39.7 milligrams per gram of base treated stem torch ginger (Figure 6).



**Figure 6:** Effect of contact time for the release of niacinamide from the adsorbent of base treated stem torch ginger. (at temperature of 30 °C).

## CONCLUSIONS

Stem torch ginger as agricultural waste can be used as a kind of natural adsorbent material for niacinamide or vitamin B3 when treated with base. This finding suggests a possible application as a new scrub in cosmetic products which can release niacinamide to the skin. This special scrub property could achieve a lighter skin color. The researchers suggest further studies, as to use other types of alkaline solutions to reduce the environmental impact such as potassium hydroxide solutions. Potassium is an essential nutrient for plant and the solutions could be finally used in agricultural applications. This could help to reduce the impact on the environment and reduce the cost of waste management.

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