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Extraction of nitrogen, phosphorus and potassium from food waste under elevated temperature by heat induced bath method

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Abstract. 38,000 tonnes of waste is generated per day in Malaysia and 15,000 tonnes of that are food wastes. This waste should be recovered to produce a new valuable product that brings benefits such as fertiliser because it contains various nutrients such as proteins, carbohydrates. This study aims to determine the effects of thermal treatment on the macronutrient of food waste that collected from cafeteria of Universiti Sains Malaysia (USM). Hot water bath was employed as thermal treatment of food waste with five different temperatures (60 °C to 100 °C) with two reaction time (30 min and 60 min). The results have demonstrated increases in the percentage of nitrogen (N) and concentration of phosphorous (P) however for the concentration of potassium (K) shows inversely related after subjected to hot water bath. Thermal treatment showed the untreated sample only consists 2.13 % of N and 31.53 mg/L of P, and after the treatment resulted in an increase in N and P by 0.22 % - 3.39 % and 0.33 mg/L - 21.40 mg/L respectively. On the other hand, the concentration of K in the untreated sample is 0.4915 ppm and after treatment it decreases to 0.1051 ppm - 0.2052 ppm. Based on the range of macronutrient in organic fertilisers in Malaysia, only N was in the range and can directly employ as organic fertiliser while for P and K should undergo further treatment.

1. Introduction

Food wastes are any food that is not consumed or discarded and generated at any level within food chain where from production, processing, distribution until consumption of food [1]. It biodegradable and the degradation of each component of food waste is affected by environmental conditions, therefore, these wastes need to dispose of properly to provide a clean and healthy environment. Food waste contains various nutrients such as natural fibers, soluble sugars, starches, fats, lipids, proteins, celluloses, and vitamins [2].

There are multi-stages of reaction associated with the production of valuable product (fertiliser and biogas), the first step is crucial and assist in increasing the production of valuable product. Therefore, pretreatment was employed because it can ensure the effective performance of further treatment. Pretreatment is a process where it produced a disrupted, solubilisation and separation of one or more components of biomass [3, 4]. It converts complex macromolecules into low molecular weight compounds [5].

Pretreatment could be categorized into physical, physicochemical, chemical, biological and combinatorial pretreatment. Thermal pretreatment is one of treatment where it involves from low to high temperature in the reaction with organic materials. The effect of thermal pretreatment are disintegration of cell membranes of food waste and resulting in the solubilisation [5].

In theory, cell lysis will performed firstly then followed by reducing the size of organic matter molecules [6].

In hot water bath, heat transfer occurs through convection then it followed by conduction. Hot water is the medium of heat transfer because it direct contact with the base of the glassware [7].

Fertiliser is any material used to increase nutrient content to the soil to promote soil fertility and increase the growth of plant. It could improve the crop yield by 50 % to 80 %. Nitrogen (N), Phosphorous (P) and Potassium (K) are the primary macronutrient in the fertiliser.

2. Methodology

2.1. Waste Collection
Food waste that used in this study was collected from Baktisiswa Cafeteria at Universiti Sains Malaysia. The organic and inorganic waste was separated and only the organic waste was used in this treatment. The segregation of waste were carried to ensure that the waste could be degradable during treatment [8]

2.2. Waste Preparation
Food waste was dried using oven dry at 105 ℃ for 24 hours to eliminate the water content then dry food waste were blended in the heavy duty blender. This is because to increase the surface area to provide better contact time thus enhances the process to breakdown the food wastes during the thermal treatment and to homogenize the composition of the food waste.

2.3. Thermal pretreatment
The powder samples were placed in a conical flask with distilled water with ratio 1:10 (W:V) where 25 g of sample with 250 ml of water [9].

Five temperatures were set up which were 60 ℃, 70 ℃, 80 ℃, 90 ℃ and 100 ℃ and two reaction times, 30 min and 60 min [6, 7]. For the control samples, food waste was placed in conical flask with distilled water at room temperature.

2.4. Macronutrient
N, P and K are macronutrients and valuable for plant nutrient. For this research, these macronutrients were analysed before and after thermal treatment

2.4.1. Nitrogen. The method that been used to analyse nitrogen was Total Khejadal Nitrogen (TKN) method [10,11]. TKN procedure basically divided into three parts which were digestion, distillation and titration.

2.4.2. Phosphorous. For phosphorous analysis, DR2800 spectrophotometer (Hach) was used [11]. The digestion of food waste was carried out first by using microwave digestion method using microwave digester (MARS 6).

2.4.3. Potassium. Potassium of the food waste were determined by using Flame Atomic Absorption Spectroscopy (FAAS).

2.5. Data analysis

The data that obtained be calculated and illustrate as graph by using Microsoft Excel. IBM SPSS is used to analyse the data by two-way ANOVA. This to identify if there is an interaction between the two independent variables on the dependent variable.

3. Results and discussion

3.1 Effect of thermal pretreatment on macronutrient of food waste

3.1.1 Nitrogen. N in food waste could be found in food which high in proteins such as meat, chicken, fish, and egg. The percentage of N that can be used as organic fertiliser was in the range of 0.7 % – 4.4 % [12] In general, as the temperature increases and longer reaction time, the percentage of N will increase. Based on Figures 1, food waste that undergoes the thermal treatment yield an increase by 0.22 % to 3.39 % in the percentage of N compared to the untreated sample. Graph with reaction time 30 min shows, the percentage of N increase at 60 ℃ and 70 ℃. However, at 80 ℃ it decreases extremely and at 90 ℃ and 100 ℃ it grew strikingly until 5.52 %. However, for the graph temperature against percentage of N for 60 min reaction time fluctuates. At 60 ℃ the percentage increase by 1.73 % then at 70 ℃ and 80 ℃ it declines to 2.72 %. Then at 90 ℃, the percentage of N increased to 4.13% and decreased back at 100 ℃.Basically, most N is either tightly bound within the solid matrix or is present in an insoluble form. After thermal treatment, increasing in N might be due to the degradation of protein molecules [13]. Protein consists of a long chain of amino acid and it is tight coils. During the thermal treatment, as the protein in food waste was heated thus the tight coils will unwind. At this point, the protein was denatured, becomes smaller and less complex chains.[14] The correlation analysis illustrate that there was no statistical significant, strong positive relationship between temperature and percentage of N for 30 min ($r = 0.571$, $p = 0.888$) while for 60 min ($r = 0.306$, $p = 0.549$) there was no statistical significant, weak positive relationship between temperature and percentage. However, as illustrated in Figure 1 the percentage of N decreasing due, incomplete digestion during digestion step in TKN analysis.
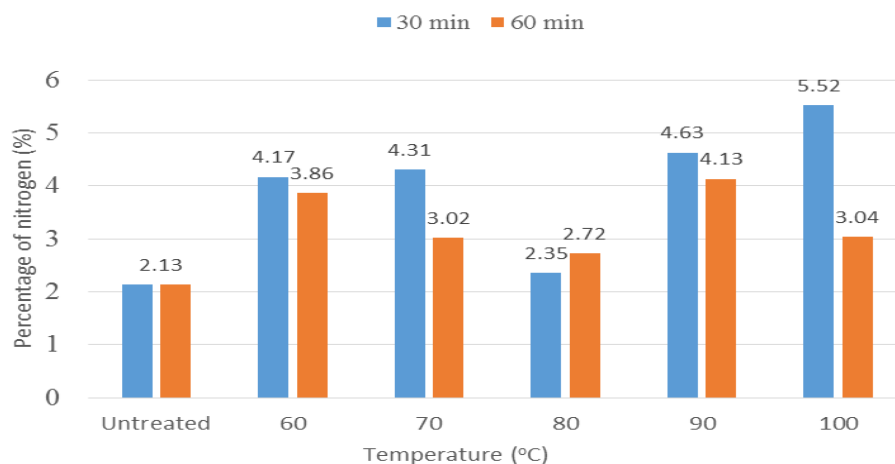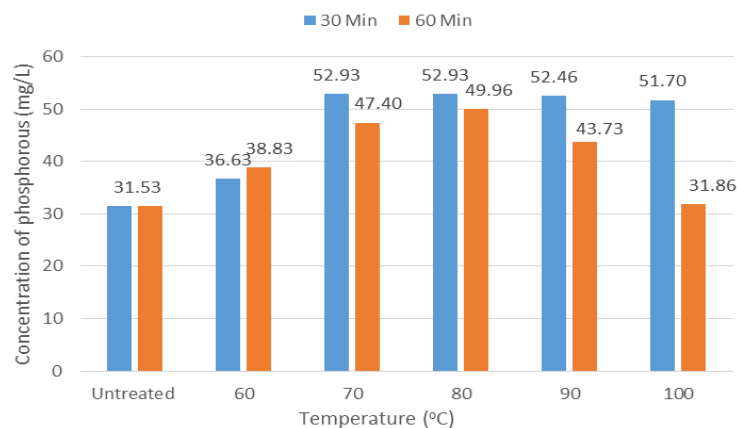

Figure 1. Nitrogen for untreated and treated food waste.

Incomplete digestion happens when not all organic bonded nitrogen was changed to ammonium ions which it indicates by the colour of the digested sample. In this condition, the digested sample was found changed in colour from black to yellowish liquid and not to clear liquid. Thus it has affected distillation and titration process thus the percentage of nitrogen fluctuated.The highest percentage of N for 30 min and 60 min reaction time is at 100 ℃ and 90 ℃ respectively. For 30 min it increases by 3.39 % and at 60 min it increases by 2% compared to untreated sample and it within range of organic

fertiliser. The reaction time for 30 min produce higher percentage of N compare to 60 min reaction time.

3.1.2 Phosphorous. Normally in food waste, P can be found in protein food (milk, meat), nuts and seeds, vegetables and fruit and whole grain. The normal range of P in organic fertiliser was 0.04 % - 8.65 % and it essential nutrient that been used for photosynthesis [12]. Higher concentration of P can be achieved with lower temperatures, but longer reaction times [5]. Figure 2 shows the graph with contact time 30 min the concentration of P is increasing to 366.30 mg/L and 52.93 mg/L as temperature increased to 60 ºC and 70 ºC. At 80 ºC the concentration of P remains constant at 52.93 mg/l. However, as the temperature starts to increase to 90 ºC and 100 ºC the concentration of P slightly decreases to 52.46 mg/L and 51.70 mg/L correspondingly.

Figure 2. Phosphorous for untreated and treated food waste.

While, graph with 60 min reaction time illustrate that as the temperature increase from 60 ºC to 80 ºC the concentration of P also increase until 49.96 mg/l but as temperature reach 90 ºC and 100 ºC the concentration drop to 31.86 mg/L. The concentration of P was increasing by 0.33 mg/L until 21.40 mg/L for all temperature and both reaction time compared to the untreated sample. Concentration of P was increasing due to the progressive disruption and disintegrate of chemical bonds in cell walls and membranes by thermal treatment [3]. Therefore, the intracellular organic material is released. At high temperature there will be a formation of insoluble phosphate. This clearly shows that thermal treatment with higher temperature will affect the concentration of phosphorous. This leads to the production of recalcitrant soluble organics hence reducing the biodegradability. The temperature of thermal treatment and percentage of P shows directly proportional with statistical significant for 30 min (r = 0.925, p = 0.000) and 60 min (r = 0.854, p = 0.042). This statistical indicate that increases in value of one variable, the second variable also increase in value. The highest concentration of phosphorous for 30 min and 60 min reaction time was at 80 ºC. For 30 min it increases to 52.93 mg/L and at 60 min it increases to 49.96 mg/L compared to the untreated sample which only 31.53 mg/L. Even though the percentage of P in food waste was found to increase due to thermal treatment but the percentage is too small which can be considered there are no P in organic fertiliser. The highest percentage of phosphorous after thermal treatment is 0.0053 % where the normal range of phosphorous in organic fertiliser is 0.04% - 8.65% [12]. Therefore, this food waste needs to undergo further treatment to increases the amount of P in organic fertiliser.

3.1.3 Potassium. The sources of K in the food sample were fruit and vegetables. 1.29% - 6.94% is the range of percentage of potassium in organic fertiliser [12]. Figure 3 shows, the concentration of K in food waste for both reaction time were decreased drastically at 60 ºC compare to untreated sample and it continuously decreases at 70 ºC. However for the graph with 30 min reaction time, the concentration gradually increase from 80 ºC until 100 ºC.
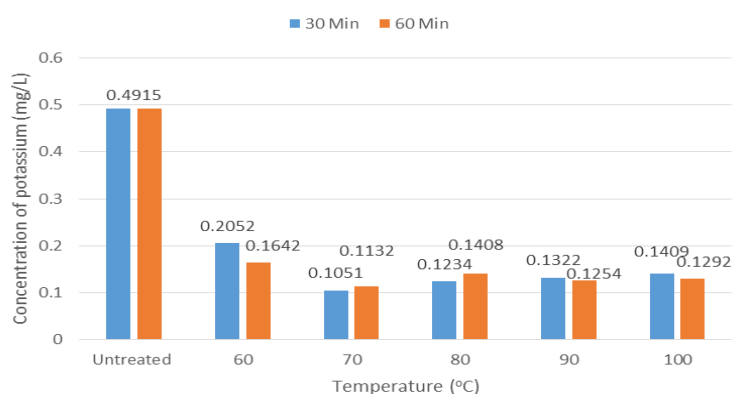
Figure 3. Potassium for untreated and treated food waste.

While for graph with 60 min reaction time illustrate that concentration of K increase by 0.0276 mg/L during 80 $^{\circ}$C but decreases slightly at 90 $^{\circ}$C and 100 $^{\circ}$C. K in food waste present in the form of potassium ions (K$^+$). During the thermal treatment, cells break down and release K$^+$ free and easily move into the water. K$^+$ are extremely soluble in water. Therefore the K$^+$ remained in the water since they were highly soluble in it [15]. The concentration of potassium at 60 $^{\circ}$C for both reaction time were higher compared to other temperature. This is because there only a small amount of cell that been break down. Therefore more K in the solid sample and only little cells break down. For reaction time 30 min the correlation analysis shows no significant, strong positive of temperature and the concentration of K (r = 0.980, p = 0.202). While for 60 min (r = 0.370, p = 0.183) there were no statistically significant, weak positive relationship. Thermal treatment using hot water bath is not suitable for potassium because this treatment could not achieve the minimum requirement of potassium for organic fertiliser. It shows decreasing in concentration of potassium compare to untreated sample and the percentage of potassium did not exist in an ideal ratio to provide nutrient for plant as organic fertiliser. Thermal treatment showed that potassium content failed to increase the level in the food waste.

3.2. Interaction between duration time and temperature by using statistical analyses

Table 1. Interaction between duration time and temperature.

Parameter	N	P	K
Temperature * time	p = 0.001	p = 0.000	p = 0.333
Temperature	p = 0.000	p = 0.000	p = 0.000
Time	p = 0.000	p = 0.000	p = 0.289

4. Conclusions
From the research, we can conclude several conclusions which are:

Percentage of N was in the range of organic fertiliser and can directly be employed. While for percentage of P its too small. Therefore it need to undergo further treatment to increase the percentage of P. However for K, hot water bath method is not suitable for potassium.

The statistical analysis two-ways ANOVA shows that there is an interaction effect between the independence variables (temperature and reaction time) and macronutrient (N,P) in food waste after thermal treatment. However, for potassium the interaction effect was not significant.

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