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RESEARCH ARTICLE

OIL AND PECTIN EXTRACTION FROM CALAMANSI LIME AND KEY LIME

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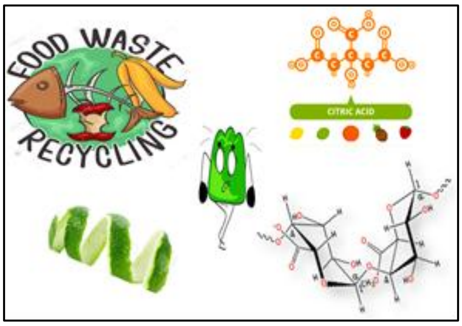
ABSTRACT

Calamansi lime and key lime are commonly grown tree fruit in the world. These limes are generally used in cooking for its amazing taste of juice. The main purpose of this study is to extract pectin from lime peel using two types of solvent which are water and ethanol. These solvents used to observe the different of chemical compounds and physical properties between both limes. DSC analyses were conducted over a temperature range from -20 °C to 200 °C at heating rate of 10 °C/min for thermal characterization. For the effect of pH, the experiment were conducted from pH 1 to pH 5. It was found that from the experimental observations that pH 1 solution gives highest yield of pectin. Key lime using ethanol as a solvent revealed highest yield at 45.7% These results demonstrate the successful extraction of lime pectin using calamansi and key lime and have potential as halal gelatine substitutes.



KEYWORDS

Halal gelatine, halal pectin, lime, limonene, terpinene.

1. INTRODUCTION

The Waste Resource Action Plan (WRAP) has estimated that 40% of food waste is untouched or unconsumed food (SWCorp Malaysia, 2019). With population of over 32 million, Malaysia generates about 38 000 metric tonnes of waste on daily basis. The highest composition of waste is the food waste. WRAP estimated that 60% of them is avoidable waste such as food leftover, rotten fruits and bread. Another 40% is unavoidable which include the fruit peels (SWCorp Malaysia, 2019).

Direct disposal of citrus waste (without proper processing) causes environment problems. Therefore, it is important to treat citrus waste systematically in food industries and other areas (Sharma et al., 2017). The citrus peels, if treated as waste materials, may create environmental problems, particularly water pollution, since the presence of biomaterials in peels such as oil, pectin, as well as sugar, stimulate aerobic bacteria to decompose the biodegradable organic matters into products such as carbon dioxide, nitrates, sulphates and phosphates in water.

Citrus fruit waste is rich in biologically active compounds including natural antioxidants, such as phenolic acids and flavonoids (Li et al., 2006). (Koubala et al., 2008). The peel, pectin extraction can be used as a high protein stock feed in dry form, increasing the potential return for fruit

Flavonoids are an important class of bioactive compounds because of their versatile antioxidant, anti-carcinogenic, and anti-inflammatory properties and their ability for lipid anti-peroxidation effects (Benavente-Garcia and Castillo, 2008). The two groups of flavonoids found in citrus fruits are glycosylated flavones (luteolin, apigenin, and diosmin glucosides) and polymethoxylated flavones.

The albedo is the main source of pectin. Pectin is a natural product of increasing importance knowing a constant growth in production and utilization (Khan et al., 2015). Pectin have many applications in food science and nutrition, cosmetics and pharmacy. They are widely used as food additives for their thickening, gelling and emulsifying properties in jams, soft drinks, fish, meat and milk products (Barrera et al., 2002).

Pectin includes all the esterified polygalacturonic acids at different degree of neutralization. In the presence of saccharine and small quantities of organic acids (usually citric acid), pectin gelatinized, and this property is exploited by the agro chemistry and pharmaceutical industries for pectin isolation (Pandharipande, 2012). Industrially, pectin is produced from citrus waste under acidic conditions at an elevated temperature of ~100°C

juice industry and reducing the pollution load to the environment (Benavente-Garcia and Castillo, 2008).

The high concentration of hydrogen ions present in the solvent (at low pH) stimulates the hydrolysis of an insoluble pectin constituent, proto pectin. Proto pectin is a compound formed by the combination of cellulose with pectin molecules. During acid hydrolysis, the combination is split up to produce soluble pectin and cellulose by eliminating water molecules (Yeoh et al., 2008). The presence of polyvalent ions such as calcium and magnesium also increases the insolubility of proto pectin. Upon treatment with acidic solvents, hydrolysis as well as the removal of calcium and magnesium ions occurs. As a result, proto pectin becomes soluble pectin.

Therefore, this study provides value added products where the lime peel waste is reuse and convert beneficial products such as oil and pectin by undergoes a few process. The pectin being further study on effect of pH extracting medium on the yield of pectin.

2. MATERIAL AND METHODS

2.1 Materials

The raw material taken under examination for the extraction of oil and pectin is lime peel. Two type of limes are used; calamansi lime and key lime. Lime peels are removed from limes and dried before blended into fine pieces. Water and ethanol are used as solvent for the extraction. Citric acid is used during acid hydrolysis in pectin extraction.

2.2 Methods

Simple distillation is employed for removal of essential oil from the lime peel. Dried and fined lime peel is added with known quantity of solvent which is simple distilled off for approximately 3 hours. The solid remains of the residue are dried to obtain the dry cake. The dried cake obtained is further treated for separation of pectin. The process flow chart is as shown in the figure 1.

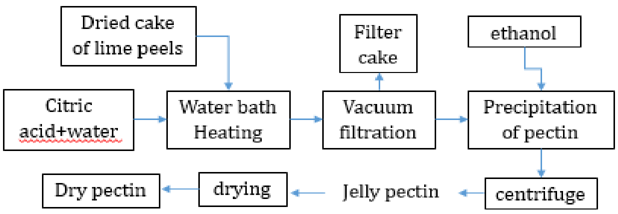


Figure 1: Process flow chart for extraction of pectin from lime peel residue

Citric acid in distilled water solutions of desired pH values 1, 2, 3, 4 and 5 are prepared. Lime peel dried residue weighing 10 gm each are dipped in to the solution and heated at 80°C for 10 minutes. After cooling the solution, it is filtered using cloth filter and Whatman filter paper under vacuum. Ethanol is added to the filtered solution to facilitate filtration of pectin. The solution is centrifuge at 4000 rpm for 15 min to separate jelly pectin which is dried at 50°C 10 hours. Dried pectin is thus obtained. Yield % of pectin is based on the gram of peel sample taken, and is calculated by formula as given below;

Yield of pectin (Ypec %) =100 x (P/Bi)

where Ypec (%) is the extracted pectin yield in per cent (%), P is the amount of dry pectin in g and Bi is the initial amount of lime peel in gram. Differential scanning calorimetry (DSC) analyses was conducted for thermal characterization. The samples were heated in an aluminium crucible at a rate of 10°C/min from -20°C to 200°C. Nitrogen was used as a purge gas with a flow rate 50mL/min (Sutar et al., 2008).

3. RESULTS AND DISCUSSION

The maximum yield of pectin is obtained at extraction medium pH of 1 for all samples. Key lime in ethanol solvent residue shows the highest percentage yield of pectin at 45.7%. However negligible yield is obtained at pH of 4 and 5 as can be seen from graph plotted between pectin yield percent obtained for various values of pH of medium as shown in figure 2.

Table 1: Condition and result for maximum pectin yield at medium pH of 1.

Type of lime peel residue	Calamansi water solvent	Calamansi ethanol solvent	Key lime water solvent	Key lime ethanol solvent
Volume of water(mL)	100	100	100	100
Amount of peel sample added (g)	10	10	10	10
Extraction temperature (°C)	80	80	80	80
Extraction time (minute)	10	10	10	10
Volume obtain after filtration (mL)	80	75	38	50
Volume of ethanol added (mL)	40	37.5	19	25
Centrifuge rpm	4000	4000	4000	4000
Centrifuge time (minute)	15	15	15	15
Weight of dried pectin obtained (g)	1.28	1.08	2.44	4.57
Percentage yield of pectin (%)	12.8	10.8	24.4	45.7

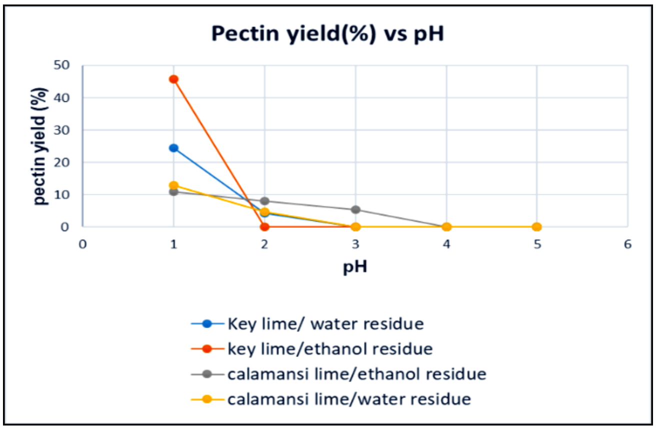


Figure 2: Pectin yield at different pH of extracting medium

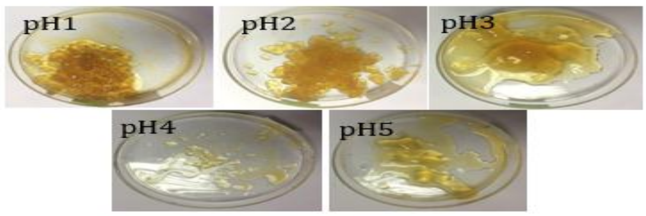


Figure 3: Jelly pectin at different pH of extracting medium

Figure 3 shows the jelly pectin obtained after centrifuging. It shows that the yield of pectin obtained could be affected by the pH value as medium extracting. Therefore, in the process of oil and pectin extraction from limes peel, it is recommended on basis of results obtained, that to first extract oil using simple distillation and then isolate pectin with acid hydrolysis technique by using lowest pH in order to obtain higher yield of pectin.

From the DSC curve profile of pectin, the glass transition (Tg) was observed. Tg is the change in heat capacity at the glass transition which is a measure of the amount of amorphous phase in the sample.

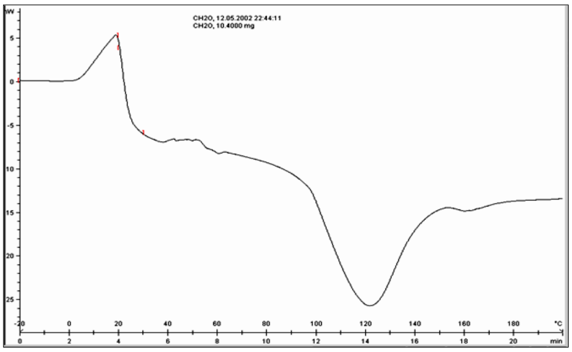


Figure 4: DSC of calamansi lime pectin from water solvent residue

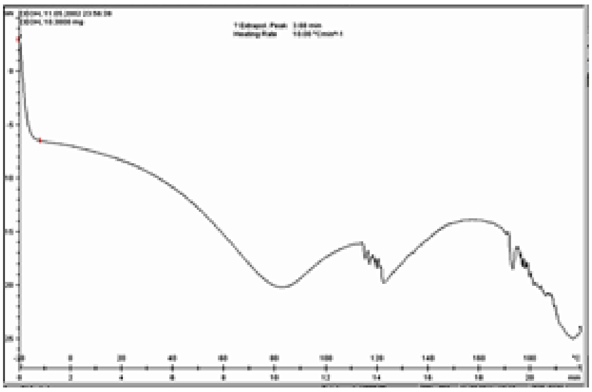


Figure 5: DSC of calamansi lime pectin from ethanol solvent residue

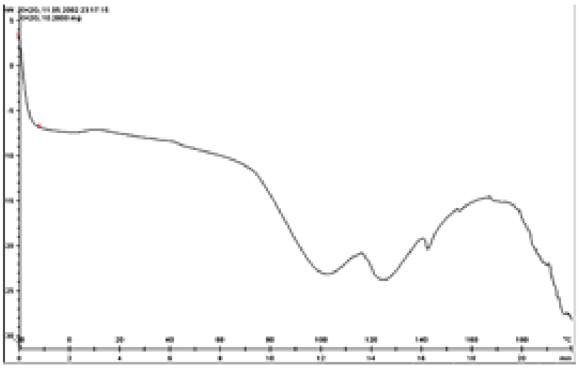


Figure 6: DSC of key lime pectin from water solvent residue

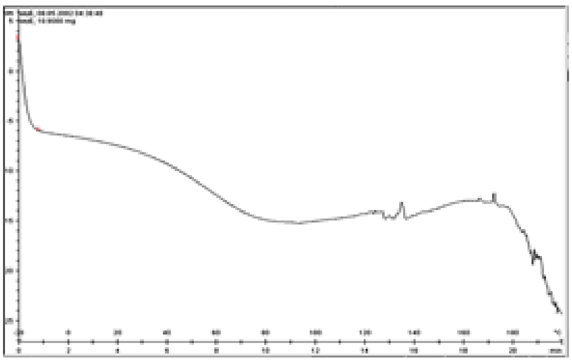


Figure 7: DSC of key lime pectin from ethanol solvent residue

From figure 4 to figure 7 indicated that the Tg are at 30°C, -10°C, 10°C and -10°C respectively. Differently from both type of key lime pectin (figure 6) (figure 7), and calamansi lime pectin from ethanol solvent (figure 5) that showed secondary or decomposition reactions that distort the peak shape after melting, the profile of calamansi lime pectin from water solvent residue (figure 4) revealed an exothermic peak at 20°C which attributed to crystallization before melting and an endothermic peak at 120°C related to process of evaporation.

4. CONCLUSIONS

Calamansi lime and key lime are commonly grown tree fruit in the world. These limes are generally used in cooking for its amazing taste of juice. Despite treating the rest as waste, the present work is dedicated for accomplishing beneficial products while providing citrus peel waste management. This help to sustain environment and save cost of composting. It is found from the experimentation that the peel source, for extraction of pectin, when taken after extracting lime oil through simple distillation gives higher yield at pH 1. So it can be concluded that the process in which lime oil is first extracted using technique of simple distillation followed by acid extraction of pectin is suitable for industrial production. These results demonstrate the successful extraction of lime oil and pectin, providing benefits from an economic and environmental point of view. Last but not least, pectin also provides potential benefit as halal gelatine in food, cosmetics and pharmaceutical industries.

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