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PRODUCTION OF PECTIN AS A RELEVANT TOOL FOR BY-PRODUCTS MANAGEMENT RESULTING FROM FOUR TROPICAL EDIBLE FRUITS PROCESSING: EXTRACTION YIELD, PHYSICOCHEMICAL AND FUNCTIONAL PROPERTIES OF PECTIN POWDER

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ABSTRACT

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Keywords

By-product valorization Ivorian fruit Pectin powder Physicochemical properties Functional properties. The appropriate re-use ways of fruit processing by-products remains a central concern for scientist and industry, especially in the current context of circular economy. Thus, this study aimed to investigate the valorization of four most processing fruit peel (orange peel, lemon, grapefruit, and avocado) into pectin as well as compare to commercial ones. The pectin yield and their physicochemical properties including ash content, water content, pH, titratable acidity and functional properties, in term of solubility and viscosity, were determined. The increasing fraction of lemon pectin was incorporated to two garden soil (black and arable) and their water holding capacity were also evaluated. The hydrochloric acid extraction method was performed to extract pectin. The results revealed that lemon peel pectin yield (25.80 %) was higher than those of orange (19.1%) followed by grapefruit (16.36%) and avocado (2.93%). Furthermore, commercial and orange peel pectin showed the highest (43.01±1.06 g/100g w.b) and the lowest (13.33 \pm 0.31 g/100g w.b) moisture contents respectively. In addition, orange peel pectin had the highest pH (2.98) similar to that of commercial pectin (3.00). Moreover, grapefruit and orange peel pectin demonstrated the highest water solubility values (≈ 55 g/100g w.d) while lemon pectin was found to be the most viscous (1.50±0.02 mPa.s) and avocado the less viscous (0.11±0.01 mPa.s). In addition, when incorporated to garden soils, lemon pectin increase soils water retention capacity. This trend was found to be high for black soil.



Contribution/Originality: This study is one of the few studies which have investigated the adding value appropriate technique for four most processing fruit by-products through pectin extraction. It also demonstrates that mixing the extracted pectin to garden soil enhance their water retention capacity, thus avoiding drastic losses during watering.

1. INTRODUCTION

Fruits and vegetables remain the major ingredients in human daily diet. However, although currently available technologies might ensure the availability into fresh state throughout the year, they are industrially processed into derived products (juice, beverage, squash and syrups) resulting in large amounts of by-products [1, 2]. For instance, by-products from citrus processing industries was estimated to be 50% of total weight [3]. Fruits industrial by-products mainly composed of peel, pulp, seeds, and whole fruits that do not reach the quality requirements, are considered as waste [2, 3]. These wastes from the European agri-food industries are estimated at more than 37 million tonnes [4] and are generally discarded out from value chain which lead to environmental pollution issues and economic losses [2, 3]. The appropriate re-use ways of these by-products remains a central concern for scientist and industry, especially in the current context of circular economy [3, 5]. Therefore, fruit industrial by-products might serve as sources of others high valuable plant-based compounds for use as ingredients in various applications [5]. Among these valuable compounds, polysaccharides have employed to design of original materials due to their various applications in many technological fields such as food, medicine, engineering, and agriculture [6]. They also demonstrated GRAS advantageous properties such as nontoxicity, biodegradability, low cost, and high degree of hydrophilicity [7].

Among various polysaccharides, pectin has shown as an interestingly known valuable plant-based polysaccharide due to their widely used as functional ingredients in feed compositions and pharmaceutical industries and agriculture [8]. Pectin refer to an anionic polysaccharide complex composed of poly- α -1-4-galacturonic acids partially acetylated or esterified by methyl groups, found in the wall of plant cells where it acts as a glue between plant cells [7, 9]. In addition, according to the Future market insights study, global pectin consumption was nearly 34,000 metric tonnes in 2016 and would reached 48,735 metric tonnes by the end of 2026 [10]. Furthermore, pectin has widely used as technological adjuvants in the cosmetic, plastic, pharmaceutical industries, medical, but especially in the food industry where it is used as a texturing gelling agent, stabilizer, thickener and emulsifier [11]. Besides, due to outstanding properties of pectin, new applications of pectin are increasingly being developed in the agricultural fields. Guilherme., et al. [8] and Sharma, et al. [7] used pectin to prepare a superabsorbent hydrogels as reservoirs to hold water and nutrients and to release them during periods of deficiencies or to avoid drastic losses during irrigation.

Pectin is industrially recovered from apple pomace and citrus peels [12]. However, others sources including sugar beets, sunflower heads, cocoa peel, potato pulp, soybean shell [13] have been investigated. Accordingly, various extraction techniques are applied to recover pectin from plant by-products including chemical, physical and enzymatic techniques Panouillé, et al. [14]. Alamineh [15] recent report has proved that acid extraction method offers a less costly, simple and successful recovery operation by providing a high yield and good properties of pectin. In addition, acid method are used for commercial pectin production [16]. It consists of hydrolysing protopectin to pectin acid in a heated acid solution, followed by precipitation with alcohol [12]. However, numerous studies focused on pectin extraction showed that the yield, physicochemical and functional properties (acid type, temperature) and extraction method [11, 12, 16, 17]. Thus, the present study aimed to extract pectin from various fruit by-products (orange, lemon, grapefruit and avocado) and to determine the yield, physicochemical and functional and functional properties of powdered pectin. In addition, the effect of pectin incorporation on two garden soils water retention capacity was also investigated for agricultural application concern.

2. MATERIAL AND METHODS

2.1. Sample Collection and Preparation

Experiment were carried out on mature lemon, orange, grapefruit and avocado peels from the experimental field of the Institut National Polytechnique Houphouët Boigny, Yamoussoukro, Côte d'Ivoire. After picking, fruits were immediately transported to the laboratory. They were carefully sorted, washed in bleached water for 10 min and rinsed with clean water. Fruits were then manually peeled and these peels were underwent blanching at 100°C for 5 min for enzyme inactivation and microorganism destruction. After cooling at room temperature, fruit peels were carefully sliced into 5 mm pieces with a stainless steel knife and oven-dried (MEMMERT) at 50°C for 48 hours as described [12]. The dried peels were then ground using a domestic electric grinder (Blender-STPE- 1220; China) and then sieved with a 1 mm mesh sieve. The coarse fraction was ground and sieved again until particles size was less than 1 mm. Finally, the powders, consisted of the fraction sum, were packaged in a high density polyethylene bag and stored in a hermetically sealed jar at ambient temperature until further analyses.

2.2. Dried Purified Pectin Powder Preparation

2.2.1. Purified Pectin Extraction Method

Pectins were extracted based on the extraction method described by De Moura, et al. [12] and Kratchanova, et al. [18] with some modifications Figure 1. Briefly, the previous prepared powder (10g) was dissolved in 200 ml of HCl (0.1 N) and heated continuously at 90°C under stirring for 45 min in a reflux system. After immediate vacuum-filtration on a whatman paper of 0.45μ m pores and icy cooling, equal volume of 96°C ethanol was added to the filtrate and left to stand for 30 min. After precipitation, precipitated pectin was separated under the same filtration condition and then washed twice with acidic alcohol (55% alcohol + 6.5% Hcl + 38% distilled water) and once with pure acetone to remove HCl and alcohol traces. The raw pectin obtained in these conditions was then dried in a ventilated oven at 50°C for 24 hours, ground for 30 seconds and stored as previous described in section 2.1.



Figure-1. Diagram of dried purified pectin preparation extracted from fruit residues.

2.2.2. Pectin Yield Determination of Different Fruits

Pectin yield were calculated by the ratio between the weight of the dried purified pectin powder resulted from the extraction process and the weight of the ground dried fruit peel submitted to the extraction according to the formula.

Pectin yield (%) = $\frac{\text{Weight of dried purified pectin powder}}{\text{Weight of the ground dried fruit peels}} \times 10$

2.3. Determination of Physicochemical Properties of Pectin Powders

2.3.1. Moisture and Ash Content

Moisture content (MC) was determined using the method described by Doymaz, et al. [19]. A test sample (1g) was dried in a ventilated oven (Memmert, Germany) at 105°C until constant weight. MC was then calculated using the followed equation.

$$MC = \frac{M_1 - M_2}{M_1 - M_0} \times 100$$

where MC denoted to the moisture content (%), M_0 , M_1 and M_2 were initial weigh of dish (g), initial weigh of powder and dish (g) and equilibrium weigh of powder and dish (g) respectively.

Ash content (AC) was determined using AOAC [20] method. Pectin sample (2g) was ashed into preweighted beaker (M₀) in a muffle furnace (Heraeus electronic, France) at 600 °C for 4 hr. After cooling into a desiccator (M₃), AC was calculated using the following equation. Each result is expressed as an average of three tests.

Ash content =
$$\frac{M_3 - M_0}{W} \times 100$$

Where W was the weight of dried matter of the test sample.

2.3.2. Determination of pH and Titratable Acidity (TA)

pH was determined using AOAC [20] method with some modification. For this purpose, 1g of pectin powder was dissolved into distilled water (50 mL) under magnetic stirring. pH was then measured using HI991001 model pH meter (Germany). Each experiments was done in triplicate. As far as concern titratable acidity (TA), the titration method according to Bamba, et al. [21] was performed with some modifications. To pectin powder (1g) placed in an erlenmeyer was added 50 ml of distilled under magnetic stirring until complete dissolution. After vacuum filtration, 10 mL was titrated with 0.1N NaOH under magnetic stirring until persistent pink color of phenolphthalein used as indicator. Each trial was carried out in triplicate. The followed equation was used to calculate TA (g/100 g DM).

$$TA = \frac{0.1 \times V_{NaOH}}{M} \times 1000$$

where M denoted to the dried weight of sample (g); V_{NaOH} was the poured volume of NaOH poured (mL); 0.1 was NaOH Normality.

2.4. Determination of Functional Properties of Pectin Powder 2.4.1. Water Solubility Test of Pectin Powder

Pectin water solubility was assessed using the method described by Bamba, et al. [21] with a slight modification. In bref, 1 g sample of pectin powder was dissolved in 100 mL of distilled water under magnetic

agitation for 20 min. After centrifugation (HETTICH model EBA III, Germany) of the mixture at 4,000 rpm for 5 min, resulted supernatant (25 mL) was oven-dried (Memmert, Germany) at 105°C until constant weight. The experiment was carried out in triplicate. Pectin water solubility was then calculated from the equation and expressed as g/100g dry matter.

Water solubility =
$$\frac{M_{sc}}{0.25 \times M_s}$$

where Msc denoted to dry weight of 25 mL(g) and MS was the weight of the supernatant (g).

2.4.2. Water Relative Viscosity Measurement Test of Pectin

Pectin powder water viscosity was determined by recording the flow time of the pectin solution using the capillary viscometer Ubbelohde (0.53) at room temperature according to the method described by Kar and Arslan [22] and Pagan, et al. [23] with some modification. The pectin solution was prepared by dissolving under magnetic agitation 0.2g of pectin powder in 100 mL of distilled water containing 5 mM Na2EDT and 0.155 M NaCl at pH adjusted to 5.0 with acetic acid to avoid aggregation of pectin molecules. After standing for 12 hours at room temperature, the mixture was filtered and 15 mL was piped into the viscometer. The viscosity was measured by measuring the flow time of the solution from a higher level to the lower level against a blanck consisted of distilled water. The density of the solution and the control were calculated by the ratio of their gravimetric weight to their volume. Viscosity was subsequently calculated using the following formula:

$$\eta = \frac{dt}{d_0 t_0} \eta_0$$

Where η (Pa.s), d (kg/m³), t (s) denote to viscosity, density and flow time of pectin solution respectively and η_0 (Pa.s), d₀ (kg/m³), t₀ (s) denote to viscosity, density and flow time of distilled water respectively.

2.5. Evaluation of the Effect of Pectin Powder on Garden Soil Water Holding Capacity

To evaluate the effect of pectin on soil water holding or retention capacity (SWHC), two kind of garden soil were used, black soil and arable soil having different characteristics.

The black soil was consisted of arable soil to which humus, NPK fertilizer were added to improve its fertility while arable soil did not contain addition. These soils were provided by the house of gardening. For this purpose, increasing pectin fraction (0, 25, 50, 75 and 100g) were carefully incorporated to each soil (50g) under agitation in a beaker. After pouring the pectin-soil properly mixture into a funnel (base being closed with wet cotton) placed on a volumetric flack, the mixture was evenly sprayed with clean pure (100 mL) and stood for 1 hour in order to collect the leached water. Each test was carried out in triplicate. Soil water holding capacity was calculated according to the following equation.

SWHC (%) =
$$\frac{V_i - V_f}{V_i} \times 100$$

Where V_i and V_f denoted to the volume (mL) of initial water and leached water.

2.6. Statistical Analysis

All the experiments were carried out in triplicate and results were reported as mean values with their standard deviation (SD). One-way ANOVA or student test were used for comparisons and statistical differences among the means were determined with Newman and Keuls post hoc tests ($\alpha = 5\%$); p < 0.05).

3. RESULTS AND DISCUSSION

3.1. Pectin Extraction Yield from Different Fruits

Pectin has been recovered from orange (*Citrus sinensis* L), lemon (*Citrus limon* L.), grapefruit (*Citrus paradisi*) and avocado (*Persea americana*) peels. The extraction yield from these fruit peels are depicted in Table 1. It can be observed that extraction yield were significantly different (p < 0.05). Lemon peel showed the highest pectin yield (25.80 ± 0.57%), followed by orange peel (19.10 ± 0.23%), grapefruit peel (16.36 ± 0.37%), and the lowest pectin yield was found from avocado peel (2.93 ± 0.19%). These results were consistent to the previous studies on plant-based pectin indicating that pectin yield was source dependent such fruit type, fruit variety and fruit organ [11, 12, 16, 17, 24]. In addition, Salma, et al. [25] reported that pectin yield of citrus peel in descending order were ranged from lemon, orange to grapefruit peel.

Fruit type	Pectin yield (%)	Moisture content (% w/w)	Ash content (% w/w)	рН	Titratable acidity (mEq g/100 g DM)
Orange	$19.10 \pm 0.23^{\rm b}$	$07.83 \pm 0.15^{\rm d}$	$4.02\pm0.27^{\rm b}$	$2.98\pm0.06^{\rm a}$	$21.67\pm0.22^{\rm d}$
Lemon	$25.80\pm0.57^{\rm a}$	$10.61 \pm 0.10^{\circ}$	$2.10 \pm 0.21^{\circ}$	$2.51\pm0.05^{\rm b}$	42.61 ± 0.28^{a}
Grapefruit	$16.36 \pm 0.37^{\circ}$	$15.67\pm0.82^{\rm b}$	$3.87\pm0.37^{\mathrm{b}}$	$2.39 \pm 0.02^{\circ}$	$25.02 \pm 0.25^{\circ}$
Avocado	$2.93\pm0.19^{\rm d}$	$16.02 \pm 0.30^{\rm b}$	7.33 ± 0.31^{a}	$2.56\pm0.09^{\rm b}$	$29.11 \pm 0.62^{\rm b}$
Commercial	-	10.01 ± 1.06 ^a	7.00 ± 0.20 a	3.00 ± 0.15^{a}	$13.67 \pm 0.50^{\rm e}$
pectin					

Table-1. Pectin extraction yield and its physicochemical properties with respect of different fruit types compared to commercial pectin.

Note: Values in the same column with different superscript symbols mean significant difference (p < 0.05).

On other hand, Masmoudi, et al. [26] when optimizing pectin extraction from lemon peel using ultrasound showed lower pectin yield (11.2%). While Donaghy and McKay [27] using enzymatic extraction (polygalacturonase) to extract pectin indicated 18 and 12.5% pectin extraction yield for lemon and orange peels respectively. In addition, the pectin yield value (15.79%) reported by Kute, et al. [28] from microwave extraction from orange peel was lower than those in this study. This could be due to high extraction time used in this study which allowed greater extraction of pectin. Moreover, De Moura, et al. [12] reported similar but slightly lower pectin yield (17.96%) from orange pomace. As regards grapefruit, similar trend of pectin yield (15.76%) was reported by Polanco-Lugo, et al. [29] using acid extraction method. Furthermore, information on avocado pectin in literature is rare. However, the report of Eaks and Sinclair [30] revealed that avocado pectin content was very low around 0.7 to 1.5%, which may explain this information scarcity. However, except lemon pectin, pectin yield value of others fruits was below the range (20-30%) reported for commercial pectin from citrus peels [29]. Nevertheless, pectin extraction yields in our study seem to be high compared to those reported in the literature. This could be due to synergic effect high extraction solution temperature, long extraction time, small particle size of the powder and the blanching before performing extraction process used in this study. Heating acid extraction solution led sufficient hydrolysis of all pectic compounds (protopectin) in cell wall resulting in their solubilisation, thus increased the yield of pectin $\lceil 31 \rceil$.

In addition, blanching favored weakening of cell wall which enhance the pectin solubilisation during extraction process [32]. In addition, powdering peels decreased particle size but increased surface exchange contact between plant material and extraction solution, leading to high mass transfer increasing in pectin solubilisation [29, 33] thus the increase in extraction yield occurred. Furthermore, pectin quantity and quality was also affected by various extraction factors such as extraction solvent, extraction time, pH, raw material, material variety and maturity stage, and pectin drying temperature [22, 34]. According to Kratchanova, et al. [18] hydrochloric acid is the best agent for high yield. However, although hydrochloric acid method is adopted for commercial pectin production due to its efficiency, it is likely to cause environmental issue resulting from the discharge of acid effluents which the industrial will still have to treat.

3.2. Physicochemical Properties of Pectin from Different Fruits

3.2.1. Moisture Content of Dried Purified Pectin Powders

Table 1 shows moisture content of pectin powder extracted from orange, lemon, grapefruit and avocado and then compared to commercial one. It can be seen that moisture content varied significantly (p < 0.05) between fruit except grapefruit and avocado pectin powder. Moisture content value was ranged from 10.61 to 16.02 % wet basis. The highest moisture content was found from grapefruit (15.67 \pm 0.82 %) and avocado (16.02 \pm 0.30 %) pectin powders which were not significantly different, greater than those of the lemon $(10.61 \pm 0.10 \%)$ and followed by orange pectin (07.83 \pm 0.15 %). When extracted pectin were compared to commercial pectin, it can be observed that commercial showed the moisture content of 10.01 ± 1.06 % Table 1. The low moisture content of 8.60 % were reported for red grapefruit peel pectin [35]. The difference with our result could be due to the fact that pectin was extracted from alcohol insoluble solid fraction of grapefruit peel while in our study, it was extracted from the whole peel. As far as we know, no data has previously been reported on moisture content of avocado peel pectin. In addition, as regards lemon pectin, Salma, et al. [25] and Azad, et al. [13] reported slight high moisture content of 12.2 and 13.40 ± 0.79 for peel and pomace respectively. Moreover, the moisture content of orange peel pectin reported in literature were variable but higher than those observed in our study. Thus, Güzel and Akpınar [36] and Srivastava and Malviya $\lceil 1 \rceil$ indicated the values of 10.18 ± 1.14 % and 22.51 % respectively. Conclusively, it should be noted moisture contents indicated in literature are highly variable for the same plant material used for pectin extraction and from one plant material to another regardless of the extraction method used [37]. However, values reported in this study seem to be slightly high as pectin is highly hygroscopic which could promote the growth of micro-organisms and the production of pectinated enzymes leading to further affected pectin quality [38]. For this reason, these pectin should be preserved in closed dry atmosphere $\lceil 25, 39 \rceil$.

3.2.2. Ash Content of Dried Purified Pectin Powders

The ash content is an important indicator of pectin purity, as pectin is likely to be pure when its ash content is low [13]. As can be seen in Table 1, ash content values varied significantly (p < 0.05) from one fruit peel to another and were ranged from 2.10 to 7.33 \pm 0.31%. The highest value was observed from avocado peel pectin (7.33 \pm 0.31%), followed by orange (4.02 \pm 0.27) and grapefruit peel pectins (3.87 \pm 0.37%), while the lowest value was shown for lemon. These values were found to be low compared to the value of commercial pectin (7.00 \pm 0.20 %) except those of avocado. Fruit ash contents were found to be extraction method and fruit type and maturity dependent [13, 40]. Previous studies indicated various values according to fruits from which pectin is recovered. Sulieman, et al. [40] reported low ash content of 0.32, 0.35 and 0.36 % for commercial, lemon and orange peel pectin respectively, while Salma, et al. $\lceil 25 \rceil$ observed lower ash content (3.3%) for lemon than those of orange peel ranged from 6.5 to 8.9%. In addition, Azad, et al. [13]; De Moura, et al. [12]; Mohamed [35] and Güzel and Akpinar [36] showed ash values of 4.06 \pm 0.29, 3.26, 1.19 \pm 0.06 and 2 % for lemon pomace pectin and orange and grapefruit peel pectin respectively using alcohol precipitation method. Likewise, Tyagi [41] reported the increase of ash values from 5.6 to 9 % for orange peel pectin with the increasing in extraction temperature from 40 to 100°C. Alternatively, Dennapa, et al. [42] showed higher ash value (22.19%) for pectin recovered from Carica papaya pulp using aluminum chloride precipitation than those obtained by alcohol precipitation (5.22%). This suggest that the ash content of pectins varied according to the plant materials and the extraction methods [36]. In addition, May, et al. [43] revealed using aluminum salts to precipitate pectin from plant fruit led to high ash content resulting from high impurity contents while pectin precipitated with alcohol was less laden with impurities, which may justify the low values obtained in this work. Furthermore, as reported by Salma, et al. [25] and Azad, et al. [13] that highquality pectin in terms of gel formation must have ash content below 10 %, the low ash content observed in this work suggest that the pectin obtained have good quality.

3.2.3. pH and Titratable Acidity of Dried Purified Pectin Powders

The pH is one of the most crucial parameters which determines microbial proliferation and therefore the product's suitability for preservation [44]. Table 1 also shows the pH and titratable acidity of 2% pectin solution. It can be observed that pectin solutions were acidic and the pH values were significantly different and ranged from 2.39-2.98. The highest pH value was found with orange pectin (2.98 ± 0.06) which was similar to the value observed with commercial pectin (3.00 ± 0.15) while the lowest pH was observed with grapefruit pectin (2.39 ± 0.02). In addition, lemon and avocado pectin solution showed similar pH value which were are values: 2.51± 0.05 and 2.56 ± 0.09 respectively. Similar results were reported by Sulieman, et al. [40] who demonstrated that pH value of orange peel (4.2) and commercial (3.9) pectins were higher than the pH value of lemon peel pectin (3.5). In addition, Bagde, et al. [45] found orange pectin pH value (4.5) higher than those of lemon pectin (3.9). Likewise, Srivastava and Malviya [1] indicated high pH value of 4.15 for 1% solution of orange peel pectin. However, previous pH values indicated above were found to be acidic but slight higher than those observed in this study. This can be attributed to pectin solution concentration which two times high in this study. Nevertheless, since pH values were under 4.5, the control of pathogenic bacteria growth is favored during storage [21].

The titratable acidity (TA) result presented in Table 1 exhibit a significant variation between plant materials (p < 0.05). Apart commercial pectin which demonstrated low TA value (13.67 \pm 0.50 mEq g/100 g DM), the TA values of extracted pectins in this study were higher than 20 mEq g/100 g DM. The highest TA was observed with lemon peel pectin (42.61 \pm 0.28 mEq g/100 g DM) followed by avocado peel pectin (29.11 \pm 0.62 mEq g/100 g DM), grapefruit peel pectin (25.02 \pm 0.25 mEq g/100 g DM) and then orange peel pectin (21.67 \pm 0.52 mEq g/100 g DM). No work have previously reported data on the titratable acidity of pure pectin solution apart from those reported on jam and jelly made with a small quantity of fruit pectin. Thus Sulieman, et al. [40] indicated low TA value of 0.38-0.49 ascorbic acid equivalent on jams made with containing 0.4% of orange pectin, lemon and commercial pectin. In addition, Ahmed and Sikder [46] showed TA values from 0.91 \pm 0.03 to 1.09 \pm 0.06% for pineapple jelly made with 1% pectin from three varieties of lemon (ginger lemon, cardamon lemon and china lemon). Since pectin is used in small quantity in food preparation, the high value obtained could not adversely affect consumers.

3.3. Functional Properties of Dried Purified Pectin from Different Fruits

Functional properties, consisting of various properties including solubility, viscosity, etc., are crucial in and for material formulation as they allow to predict its fate. They explain how an ingredient can behave during preparation and how it may impacts the finished product appearance, texture, structure and taste [47]. Thus, the functional properties of material depend on their components and the structures of these components as well. Therefore, pectin powder may impart its specific properties to non or food system during the rehydration process, only if it is able to interact with water. The results of water solubility, referring to the ability of pectin powder to dissolve in water, are presented in Figure 2a. As shown, pectin powder water solubility was significantly influenced by fruit type. The highest solubility was recorded with pectin from orange (55.33 \pm 0.81 g/100g DM) and grapefruit (56.01 \pm 0.70 g/100g DM) peels while the lowest solubility was found with peetin from lemon peel $(29.12 \pm 1.15 \text{ g}/100 \text{g} \text{ DM})$. This result is consistent with those reported [48] who demonstrated significant water solubility difference between pectin from papaya, mango and banana. However, their solubility values reported was higher (above 85.36 ± 0.46 %) than those in this study. The difference would be attributed by the analytical methods used to assess the solubility and likewise the raw material. Above mentioned authors performed solubility test into hot water whereas we used tepid water (25°C). In addition, previous works reported that pectin from lemon, grapefruit and orange peels dissolved in easily hot water and were insoluble in cool water except orange pectin which was slight soluble in cool water, however, they were insoluble in organic solutions [36, 41, 49]. Furthermore, although pH did not significantly affect pectin water solubility [50] water solubility is highly affected

by the degree of esterification of pectin due to the hydrophobic nature of esters with long hydrocarbon chains [51]. The higher the degree of esterification, the lower the water solubility [50, 51]. This suggest that the relatively low solubility found in this study may be due to its middle degree of esterification.



Figure-2. Water solubility (a) and viscosity (b) of dried purified pectin powders of different fruits.

On the other hand, Figure 2b shows the results for relative viscosity of pectin extracted from different fruits in water. It can be noticeable that the source of pectin significantly (p < 0.05) affect pectin powder water viscosity. The highest value of water viscosity was observed with lemon peel pectin (1.54 ± 0.02 mPa.s), followed by orange pectin (1.02 ± 0.05 mPa.s), grapefruit pectin (0.81 ± 0.03 mPa.s) and lastly avocado pectin (0.11 ± 0.02 mPa.s). These results were consistent to those reported by Sayah, et al. [52] when studying on relative viscosity with degree of space-occupancy for orange and grapefruit pectins. Furthermore, Aida and Norasmanizan [48] also observed a significant difference by comparing the intrinsic viscosity of mango peel pectin, banana peel pectin and papaya peel pectin. In addition, Pacheco, et al. [53] also reported that pectin extracted by enzymatic method showed higher apparent viscosity than pectin extracted by acid method when using sugar beet peel. They also argued that dried pectin showed lower apparent viscosity than pressed and ensiled pectin from sugar beet pulp, regardless the extraction method. These results therefore suggest that viscosity would depend not only on the raw material type and state but also on the extraction methods. This could explain the relative low relative viscosity found in this

study as higher extraction temperature lead to a greater depolymerisation of pectin into shorter chain, and consequently in lower viscosity [54]. Furthermore, lemon pectin, which exhibited low solubility, had the highest viscosity, suggesting lemon pectin present higher esterification degree than others pectins in this study as previously mentioned in solubility section. Indeed, viscosity which denote to the capacity of the solute to increase the viscosity of the solution by gel formation [48] is caused by hydrogen bonding between free carboxyl groups on the pectin molecules and also between the hydroxyl groups of neighbouring molecules [55].

3.4. Effect of Pectin Powder on Garden Soil Water Holding Capacity (WHC)

The section demonstrated the applicability of pectin in the management of water in agriculture in lab-scale. This experiment was conducted with pectin extracted from lemon which has shown the highest viscosity. The results of pectin addition to soil on their WHC are shown in Figure 3. It can be seen that at time zero, which corresponds to the absence of pectin in the soils, the WHC were 21.12 ± 1.91 and 30.66 ± 2.13 % for black soil and arable soil respectively. When pectin was added to the soil (0.5 %), the WHC were enhanced by 52.33 ± 2.56 and 65.32 ± 1.52 % for a able and black soil respectively, corresponding to a difference in WHC of 12.99% between arable and black soils. In addition, further increase in pectin ratio up 2% led to the increase in WHC regardless the type of soil used. The WHC was 60.80 ± 1.03 and $79.33 \pm 2.29\%$ respectively for black soil and arable soil using 2% pectin ratio in soil representing a rise of 174.86 and 158.74% over initial state. It is also noteworthy that the WHC for black soil remained higher than that of arable soil. These results were consistent with those of Bahaj, et al. [56] when incorporating increasing fraction of super-absorbent polymers in soil. Likewise, Sharma, et al. [7] have previously demonstrated that soil mixed with pectin based-nanoparticle contributed to retain water for long time compare to reference native soil, providing plant growth favorable condition. This could be due to swelling capacity of pectin by holding water on their surface by sorption or trapping in microcapillaries or interparticle voids because of the greater number of free hydroxyl group in the pectin structure and also their particle properties $\lceil 29 \rceil$. These results illustrate the important role of polymers such as pectin in improving soil moisture. Furthermore, the lower water retention capacity of arable soil is likely resulted from its high porosity easing water to drain compared to black soil. However, black soil consisting of a mixture of several organic materials (humus and fertilizer), trap more water due to their porosity reduction and also the crosslinking reaction of mineral and pectin, consequently water loss is prevented [7]. Ultimately, the soil water retention capacity improvement by adding pectin observed in our study would be a strategy to prevent water loss through drainage and evaporation.



Figure-3. Change in soil water retention capacity as a function of the proportion of lemon dried purified pectin powder incorporated.

4. CONCLUSION

From this study, it can be concluded that pectin can be extracted from citrus by-product such as orange peel, lemon peel, and grapefruit peel but not from avocado which showed low pectin yield (2.93 \pm 0.19%). The pectin yield significantly differ within fruits and the highest was from lemon peel ($25.80 \pm 0.57\%$), followed by orange peel pectin (19.10 \pm 0.23%) and grapefruit peel pectin (16.36 \pm 0.37%). In addition, the physicochemical properties of extracted powder pectin were found to be significantly different within fruits. Pectin from avocado and grapefruit showed highest moisture content (16%) whereas the highest ash content resulted from avocado and commercial pectin (7%) compared to lemon pectin which was the lowest (2%). Moreover, pectin showed acidic pH which was lower than 3. The extracted powdered pectin from fruits had high titratable acidity and the highest value was shown from lemon pectin. For functional properties analysis, the highest solubility was recorded with pectin from orange and grapefruit peels (55 g/100g DM) while the lowest solubility was from lemon peel (29.12 \pm 1.15 g/100g DM). However, the highest viscosity was observed with lemon peel pectin $(1.54 \pm 0.02 \text{ mPa.s})$, followed by orange pectin (1.02 \pm 0.05 mPa.s), grapefruit pectin (0.81 \pm 0.03 mPa.s) and lastly avocado pectin (0.11 \pm 0.02 mPa.s). In addition, for application state for agriculture concerned, addition of increasing pectin ratio to black and arable soils resulted in the increase in water holding capacity regardless the soil types. Pectin behaviour as a water repellent is a promising axis for the use of pectin in agriculture to improve plant productivity and the preservation of water, soil and biodiversity resources.

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