Fakayode and Abobi *Journal of Analytical Science and Technology* (2018) 9:20
https://doi.org/10.1186/s40543-018-0151-3

Journal of Analytical Science and Technology

RESEARCH ARTICLE Open Access

Optimization of oil and pectin extraction from orange (*Citrus sinensis*) peels: a response surface approach

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Abstract

The effects of processing parameters on essential oil and pectin extractions from orange peels were investigated using response surface approach; 2 × 5 and 3 × 5 central composite rotatable designs were adopted for the essential oil and pectin extractions, respectively. Extraction temperatures (80–100 °C) and extraction times (120–240 min) were chosen for essential oil extraction, while extraction temperatures (80–100 min), extraction times (60–120 min), and extraction pH (1.0–3.0) were chosen for pectin extraction. Essential oil yield ranged from 0.57–3.24%, while pectin yield ranged from 12.93–29.05%. The predicted optimum value for essential oil yield was 3.38% at extraction temperature of 95.23 °C and extraction time of 231.30 min, while the predicted optimum value for pectin yield was 30.00% at extraction temperature of 93.07 °C, extraction time of 117.00 min, and extraction pH of 1.60. Deviations between experimental and predicted values were low and statistically insignificant. All processing factors have significant effects on both extractions. The physicochemical properties of the essential oil and pectin extracted at the optimum conditions fell within tolerable and acceptable range.

Keywords: Essential oil (EO), Pectin, Extraction temperature, Extraction time, Extraction pH, Optimization, Response surface methodology (RSM), Physicochemical properties

Introduction

Citrus represents one of the major fruits in the world. The fruits belong to six genera (*Fortunella*, *Eremocitrus*, *Clymendia*, *Poncirus*, *Microcitrus*, and *Citrus*), but the major commercial fruits belong to genus *Citrus* which consists of several important fruits, viz. oranges, lemons, limes, grapefruit, mandarins, and pomeloes (Chanthaphon et al. 2008). Orange, specifically, the sweet orange (*Citrus sinensis*) is the most commonly grown tree fruit in the world (Pandharipande and Makode 2012). Orange trees are cultivated extensively in tropical and subtropical climates due to the sweet fruit obtained. The fruits are peeled or cut (for bitter rind avoidance) and eaten skinned; or processed to orange juice by extraction. Orange fruits are majorly utilized by juice processing industries, with the peels categorized as wastes. Even at homes, orange peels are discarded inside waste container because people love the orange pulp for its juicy pulp and discard the peel as it does not taste as great as the pulp. In lieu of this, since the yield of orange juice is half of the fruit weight (Hashmi et al. 2012), a very large amount of orange by-product wastes are generated annually. Fernández-Lopes et al. (2004) reported that the by-products from orange juice processing pose a serious problem to the industry, given their limited applications and low added value. However, Nogueira et al. (2000) observed that the orange agro-industry is amongst the sectors producing large amounts of waste with possibilities for use. This recovery is absolutely connected to sustainable development and the marketability of these by-products. As there is always an increased concern about bringing useful products from waste, orange fruit wastes are no exceptions. Amongst the biologically active compounds (BAC) in these by-products are essential oils (EOs) and pectin.

Essential oils are mixtures of many compounds consisting mainly of isoprenoids, monoterpenes, and sesquiterpenes (Marin et al. 2007). They are a group of volatile aromatic compounds produced by several plant species. These BAC are responsible for the scents of many

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aromatic plants (Rossi et al. 2011; Yu et al. 2011) and can be used for pharmaceuticals, food flavor additives, and natural antimicrobials amongst others (Bakkali et al. 2008). Essential oils are utilized in the manufacture of food and medicines as flavoring agents, cosmetics, and domestic household products (Braddock et al. 1986). They also exhibit antibacterial, antifungal, and insecticidal properties (Burt 2004; Thormar 2011; and Zoubiri and Baaliouamer 2014). They are produced from orange peel extraction using several extraction methods which include steam or hydro-distillation, water, steam and organic solvent extraction, cold pressing, and supercritical CO_2 (Palazzolo et al. 2013). Essential oils from orange peels are very complex matrices which consist of many compounds of various chemical classes which are majorly separated into two parts, viz. the volatile part which constitutes between 85 and 99% and the non-volatile part which constitutes between 1 and 15% (Palazzolo et al. 2013). The oil content of citrus peels range between 0.5 and 5.0% (*w*/*v*) as reported by Palazzolo et al. (2013). They are complex natural mixtures of lipophilic substances constituting about 20–60 components at different concentrations and consist of two or three major components, namely limonene, pcymene, and ocimene at fairly high concentrations (20–70%) compared to other components which are present in trace amounts (Palazzolo et al. 2013). Several factors affect the quality, quantity, and composition of orange peel essential oil which include the nature of the fruit, provenance, genotype, soil type, climate, plant organ, age, vegetative cycle, and the extraction process (Dugo et al. 2000 and Palazzolo et al. 2013).

Pectin is produced commercially in the form of white to light brown powder mainly extracted from orange fruits. They can be produced from orange peels using several methods which include microwave, ultrasound, high pressure, subcritical water, enzyme utilization, electromagnetic induction heating, and combination of chelators like citric acid in the conventional process (Putnik et al. 2017). Pectin is a complex mixture of polysaccharides which occurs in the primary cell walls of terrestrial plants. It is composed of a linear backbone of α (1-4)-D-galacturonic acid residues which is partially esterified with methanol, with periodic interruptions to L-rhamose residues that make the backbone irregular and with some other neutral sugars present as side chains (Garna et al. 2004). Pectin consists of all the esterified polyalacturonic acids at various levels of neutralization (Pandharipande and Makode 2012). Large structural diversity is prevalent amongst pectin, chiefly because of the different plant origins and the extraction process being utilized. At present, commercial pectins are almost exclusively gotten from citrus peel, a by-product of juice manufacturing unit. The amount of pectin contained in orange peels is estimated by experiments. According to Rouse and Crandall (1976), May (1990), and Khan et al. (2015), dried citrus peel contains about 30% pectin (on dry basis). During pectin extraction from orange peels, several factors affect the process which include the pH, temperature, solvent used for extraction, time of extraction, agitation rate, and liquid solid ratio (LSR) amongst others.

Several works have been done on essential oil and pectin extractions from orange peels, though the effects of processing factors on the extractions were not considered in some. Ezejiofor et al. (2011) worked on essential oil extraction from orange peels. Also, Hashmi et al. (2012) studied essential oil and pectin extraction from sweet orange. Nonetheless, the reports did not study the influence of processing factors on the oil and pectin yields. The effects of one of the process parameters on pectin yield during extraction from orange peels have been studied by several researchers. Pandharipande and Makode (2012) worked on the separation of oil and pectin from orange peel and studied the effect of pH on the pectin yield. Njoku and Evbuomwan (2014) considered the effect of extraction time on oil extracted from orange, lemon, and lime peels. Yadav et al. (2017) also studied the effects of pH on pectin yield extraction from sweet lime peels. In the above studies, other processing factors were not considered. According to Fan et al. (2011), the traditional one-factor-at-a-time approach is time-consuming and is almost impossible to achieve the true optimal conditions; therefore, it seems unrealistic to optimize the essential oil and pectin extraction processes using that approach. Some researchers studied the effects of more processing factors on pectin yield during extraction from orange peels; howbeit, the processing parameters were not optimized. Dehankar et al. (2015) studied the removal of essential oil and extraction of pectin. For the latter, the pH, process period, and solvent used were considered but not optimized. El-Nawawi and Shehata (1987) and Rehmann et al. (2004) investigated the effects of temperature, pH, and duration on pectin extraction from orange and mango peels, respectively. However, the process was not optimized. Nevertheless, there have been reports on the optimization of processing factors on pectin extraction from other fruit peels. Kliemann et al. (2009) optimized the effects of temperature, pH, and extraction time on pectin acid extraction from passion fruit peel. Tang et al. (2011) optimized the effects of pH, extraction time, and ethanol ratio on pectin acid extraction from dragon fruit peel.

The current annual world production of oranges has been estimated to be 49.3 million tons (USDA 2018). Nigeria is ranked ninth in the world amongst the leading citrus producing countries and first in Africa, producing 3.33 million tons of citrus fruits (The Daily Records 2018). Despite this high rank, the country's local

production of essential oil and pectin is very low, as nearly 100% of the essential oils and pectin utilized by the various industries are imported. This is due to lack of appropriate and adequate data. This study therefore employs response surface methodology (RSM) to optimize the oil and pectin extraction process parameters. Development of mathematical models would be useful in predicting the responses during the essential oil and pectin extraction processes at different processing conditions, while the characterization of the essential oil and pectin at the optimum processing conditions will provide a true reflection of the quality, suitability, and potential of the extracted essential oil and pectin.

Methods

Sample preparation

Fresh oranges were purchased from the market. They were peeled, and the peels were removed, chopped into smaller pieces, and dried for 1–2 h. The dried peels were ground to give consistent and fine particles (this was essential to avoid clumping during solvent extraction) and stored at ambient temperature for further use.

Experimental design

The effects of extraction temperature and extraction time on essential oil yield from orange peels were investigated, while the effects of extraction temperature, extraction time, and extraction pH on pectin yield from orange peels were investigated. The experimental designs adopted were 2×5 and 3×5 factorial central composite rotatable design (CCRD) of response surface methodology for essential oil and pectin extractions, respectively. Central composite rotatable design consists of three types of design points, viz. factorial points (n_f), axial points (n_a), and central points (n_c). The total number of treatment combinations is $n = 2^k(n_f) + 2k(n_a) + k(n_c)$ where k is the number of independent variables and n is the number of repetition of experiments at the center point. The total number of design points is thus $N = 2^k + 2k + n_o$ (Fakayode and Ajav 2016). For essential oil extraction, 13 experiments were generated consisting of 2^2 factorial CCD, with 4 axial points ($\alpha = 2$) and 5 replications at the center points, while for pectin extraction, 20 experiments were generated consisting of 2^3 factorial CCD, with 6 axial points ($\alpha = 2$) and 6 replications at the center points. The various levels for each independent variable were selected based on preliminary experiments, observations, and previous reports by other researchers. For oil extraction, extraction temperatures (80, 85, 90, 95, and 100 °C) and extraction times (120, 150, 180, 210, and 240 min) were chosen, while for pectin extraction, extraction temperatures (80, 85, 90, 95, and 100 °C), extraction times (60, 75, 90, 105, and 120 min) and pH (1, 1.5, 2, 2.5, and 3) were selected. The dependent factors considered in this study were considered as the most important factors affecting oil and pectin yield (Gama et al. 2015; Khan et al. 2015).

Essential oil extraction

Essential oil extraction from orange peels was done using the Soxhlet method. The orange peels were pureed using a blender. A round bottom flask was washed, oven dried, and cooled in a desiccator. A dried mass of 5 g of the puree was weighed using a filter paper, and the weight recorded as W_s. The weighed sample was wrapped in the filter, tied using a thread, and dropped in the Soxhlet extractor. N-hexane was added until it siphoned once, and more hexane was added until the barrel of the extractor was half full. The condenser was checked making sure that its joints were tight, and the cooling water was circulating (the water helps to cool the extractor which is necessary to prevent heat from heating up the Soxhlet extractor, thereby resulting in equipment damage). The heating mantle was adjusted to five different temperatures (80, 85, 90, 95, and 100 °C) and left to boil gently in the round bottom flask for different durations (120, 150, 180, 210, and 240 min). After this period, the n-hexane would have completely siphoned up to the Soxhlet extractor. The setup was dismantled, and the n-hexane recovered. A beaker was washed, oven dried, weighed, and the weight recorded as W_e. The miscella (oil + hexane) in the round bottom flask was transferred into the beaker and placed inside the steam bath for 4 h to completely evaporate the n-hexane. After the n-hexane was completely evaporated, the beaker containing the sample was collected and the weight recorded as W_f. The percentage essential oil yield was calculated using Eq. (1):

$$\mathrm{EOY} = \frac{(W_f - W_e)}{W_s} \times 100 \tag{1}$$

EOY= essential oil yield (%), W_s= weight of sample (g), W_e= weight of empty flask (g), and W_f= weight of flask and extracted oil (g)

Pectin extraction

Pectin extraction from orange peels was done in two stages. Oil was first extracted from the orange peel samples after which pectin was isolated with acid hydrolysis technique as suggested by Pandharipande and Makode (2012). The inner part of the peels (albedo) contains the pectin, while the outer part (flavedo) contains d-limonene oil. Simple distillation was employed for essential oil removal from the orange peels. Twenty-five grams of dried orange peels was weighed into a steel container and blended in 1000 ml distilled water. The homogenate was transferred to a 1500 ml beaker, and the pH of the mixture was adjusted by adding

hydrochloric acid drop wisely to get the desired pH values (1, 1.5, 2, 2.5, and 3). The mixture was heated at different temperatures (80, 85, 90, 95, and 100 °C) and intermittently stirred. The pH was adjusted every 15 min, and lost water replaced. Mixture was rapidly cooled to 40 °C in an ice bath and filtered using Whatman filter paper under vacuum. The filtrate was transferred to another steel container and coagulated using equal amount of 95% ethanol and left for different durations (60, 75, 90, 105, and 120 min) to allow the pectin float on the surface. The gelatinous pectin flocculants were cleared off using a spatula, and the weight was determined. The gelatinous pectin flocculants were filtered through cheese cloth and washed with 95% ethanol to remove the remaining impurities; thereafter, it was washed with acetone drop wise and filtered again through cheese cloth. It was then dried at 60–70 °C overnight in an air-forced oven. The percentage of dried pectin yield was calculated using Eq. (2):

$$\mathrm{DPY} = \frac{W_\mathrm{d}}{W_\mathrm{p}} \times 100 \tag{2}$$

DPY= dried pectin yield (%), W_d= weight of dried pectin obtained (g), and W_p= initial weight of orange peel powder used for extraction (g).

Characterization of orange peel essential oil

The essential oil extracted from orange peels at the optimum processing conditions (the highest yield) was characterized by determining the physicochemical properties.

Determination of specific gravity of orange peel essential oil

All experiments were carried out in triplicates, and average values taken. This was determined using the procedure described by Adepoju and Eyibio (2016). A specific gravity bottle was washed, dried, and filled with water. It was weighed and recorded as W_w. The bottle was emptied and properly dried after which it was filled with the oil. It was also weighed and recorded as W_o. The specific gravity was calculated using Eq. (3):

$$\mathrm{SG} = \frac{W_\mathrm{w}}{W_\mathrm{b}} \times 100 \tag{3}$$

SG = specific gravity of orange peel essential oil, W_w = weight of specific gravity bottle filled with water (g), and W_b = weight of specific gravity bottle filled with oil (g)

Determination of free fatty acid of orange peel essential oil

This was determined as reported by Fakayode et al. (2016). Ten grams of the oil sample was accurately weighed into a 250-ml stopper flask. In a second flask, 50-ml of ethanol was heated to the boiling point, and while still over 70 °C, it was neutralized with 0.1 M potassium hydroxide using phenolphthalein indicator. The neutralized ethanol was poured in the first flask containing the oil, and the content of the flask mixed. They were boiled, and while still hot, titrated with 0.1 M potassium hydroxide, shaking vigorously during the titration. The end point of the titration was reached when the addition of a single drop of 0.1 M potassium hydroxide produced a slight, but definite color change persisting for at least 15 s. The FFA was then calculated using Eq. (4):

$$\mathrm{FFA} = \frac{28.2 \times V \times N}{W_\mathrm{o}} \tag{4}$$

FFA= free fatty acid (mg KOH/g), V= volume of 0.1 M potassium hydroxide used (ml), N= normality of the ethanolic potassium hydroxide (0.1 M), and W_o = weight of oil (g)

Determination of acid value of orange peel essential oil

This was determined using the procedure described by Njoku and Evbuomwan (2014). Twenty-five milliliters of diethyl ether and 25 ml of ethanol were mixed in a 250 ml beaker. The resulting mixture was added to 5 g of the oil in a 250 ml conical flask. Few drops of phenolphthalein were added to the mixture, and the mixture was titrated with 0.1 M potassium hydroxide and consistently shaken until a dark pink color was observed. The volume of the 0.1 M potassium hydroxide was noted. The acid value was calculated using Eq. (5):

$$\mathrm{AV} = \frac{56.1 \times V \times N}{W_\mathrm{o}} \tag{5}$$

AV = acid value of orange peel essential oil (mg KOH/g)

Determination of saponification value of orange peel essential oil

This was determined as reported by Ezejiofor et al. (2011). Two grams of the oil was weighed into a 200 ml conical flask, and 50 ml of 0.5 M alcoholic potassium hydroxide was added. This was refluxed for 1 h, followed by the addition of two drops of phenolphthalene indicator and was titrated with 0.5 M hydrochloric acid until the pink color disappears. A blank titration was equally performed. The saponification value was calculated using Eq. (6):

$$\mathrm{SV} = \frac{28.05 \times (B - S)}{W_\mathrm{o}} \tag{6}$$

SV= saponification value of orange peel essential oil (mg KOH/g), B = blank titration value (ml), and S = sample titration value (ml)

Determination of iodine value of orange peel essential oil

This was determined as reported by Takeoka et al. (1997). Three hundred milliliters of the oil was weighed and dissolved in 15 ml of cyclohexane. Twenty-five milliliters of Wijs solution was added, and the reaction was carried out in the dark for 1 h. The reaction was stopped by adding sodium iodide solution. The remaining iodine was titrated using 0.1 M sodium thiosulfate solution. The iodine value was calculated using Eq. (7):

$$\mathrm{IV} = \frac{(B - S) \times N \times 12.69}{W_o} \tag{7}$$

IV= iodine value of orange peel essential oil (mg I_2/100 mg)

Determination of peroxide value of orange peel essential oil

This was determined using the procedure described by Njoku and Evbuomwan (2014). Thirty milliliters of acetic acid chloroform solution was measured into a flask containing 2 g of the oil sample. A 0.5 ml saturated solution of potassium iodide was then added, followed closely by the addition of 30 ml of distilled water. The flask content was then titrated against 0.1 M sodium thiosulfate until the yellow color almost disappeared; 0.5 ml starch indicator was added and the titration continued until the end point (where the blue-black color just disappeared). A blank titration was equally performed. The peroxide value was calculated using Eq. (8):

$$\mathrm{PV} = \frac{100 \times (B–S)}{W_o} \tag{8}$$

PV= peroxide value of orange peel essential oil (mEq O_2/kg)

Determination of refractive index of orange peel essential oil

This was determined as reported by Adepoju and Eyibio (2016). A digital refractometer was used to determine the refractive index of the essential oil. Water at room temperature was circulated around the glass slide to keep the temperature uniform and also to normalize the refractometer. A syringe and needle were used to put few drops of oil into the glass slide of the refractometer, and the reading was recorded.

All experiments were carried out in triplicates, and average values taken.

Characterization of orange peel dried pectin

The dried pectin extracted from orange peels at the optimum processing conditions (the highest yield) was characterized by determining the physicochemical properties.

Determination of ash content of orange peel dried pectin

This was determined as reported by Yadav et al. (2017). Five grams of the dried pectin sample was put into a weighed empty crucible. The crucible was transferred to a furnace set at 60 °C to burn off all the organic matter. The carbon charred and then burnt off as carbondioxide, leaving a dark ash. This process lasted for 24 h. The crucible was taken out of the furnace and placed in a desiccator to cool. The crucible after cooling was reweighed again. The ash content was calculated using Eq. (9):

$$\mathrm{AC} = \frac{W_a}{W_s} \times 100 \tag{9}$$

AC= ash content of orange peel dried pectin (%), W_a = weight of ash (g), and W_s = weight of sample (g)

Determination of degree of esterification of orange peel dried pectin

This was determined using the titrimetric method described by Silva et al. (2008). Twenty grams of dried pectin was moistened with ethanol and dissolved in 20 ml of deionized water at 40 °C. After complete dissolution of pectin, five drops of phenolphthalein were added to the solution. The solution was thereafter titrated with 0.5 M sodium hydroxide, and the volume of the sodium hydroxide solution used for color change was recorded as V_1. Subsequently, 10 ml of 0.5 M sodium hydroxide was added, and the solution was shaken strongly and allowed to rest for 15 min. Also, 10 ml of 0.5 M hydrochloric acid was added and the solution was shaken until the pink color disappeared. The solution was titrated with 0.5 M sodium hydroxide for the last step, and the volume consumed was recorded as V_2. The degree of esterification was calculated using Eq. (10):

$$\mathrm{DE} = \frac{V_2}{V_1 + V_2} \times 100 \tag{10}$$

DE= degree of esterification of orange peel dried pectin (%),V_1 = initial titer (ml), and V_2 = final titer (ml)

Determination of equivalent weight of orange peel dried pectin

This was determined as reported by Yadav et al. (2017); 0.5 g of dried pectin was put in a 250 ml conical flask, and 5 ml ethanol was added. One gram of sodium chloride and 100 ml of distilled water were added. Finally, six drops of phenol red was added and titrated against 0.1 M sodium hydroxide. Titration point was indicated by pink color. The equivalent weight was calculated using Eq. (11):

$$\mathrm{EW} = \frac{W_s \times 1000}{V_a \times N_a} \tag{11}$$

EW= equivalent weight of orange peel dried pectin (%), V_a = volume of alkali (ml), and N_a = normality of alkali

Determination of methoxyl content of orange peel dried pectin

This was done using the Ranganna's method as reported by Yadav et al. (2017). The neutral solution was collected from determination of equivalent weight, and 25 ml of sodium hydroxide was added. The mixed solution was stirred thoroughly and kept at room temperature for 30 min. After 30 min, 25 ml of 0.25 M hydrochloric acid was added and titrated against 0.1 M sodium hydroxide. The methoxyl content was calculated using Eq. (12):

$$\mathrm{MTC} = \frac{V_a \times N_a \times 3.1}{W_s} \tag{12}$$

MTC= methoxyl content of orange peel dried pectin (%)

Determination of total anhydrouronic acid content of orange peel dried pectin

This was done using the method adopted by Yadav et al. (2017). Total anhydrouronic acid content of dried pectin was obtained using Eq. (13):

$$\mathrm{AUA} = \frac{176 \times 0.1y \times 100}{W_s \times 1000} + \frac{176 \times 0.1z \times 100}{W_s \times 1000} \tag{13}$$

AUA= total anhydrouronic acid content of dried pectin (%), y = volume of NaOH from equivalent weight determination (ml), and z = volume of NaOH from methoxyl content determination (ml)

Determination of acetyl value of orange peel dried pectin

This was done using the Ranganna's method as reported by Kliemann et al. (2009); 0.5 g of dried pectin sample was dissolved in 0.1 M sodium hydroxide solution with stirring and allowed to stand overnight. The contents were diluted to 50 ml with distilled water and an aliquot (20 ml) was placed into the distillation apparatus. Magnesium sulfate-sulfuric acid solution (20 ml) was also transferred to distillation apparatus and distilled, and about 100 ml of distillate was collected. The distillate was titrated with 0.5 M sodium hydroxide using phenol red indicator. A blank distillation using 20 ml of the magnesium sulfate-sulfuric acid solution was carried out, and the distillate was titrated. The acetyl content was calculated using Eq. (14):

$$\mathrm{ACV} = \frac{V_a \times N_a \times 4.3}{W_s} \tag{14}$$

ACV= acetyl value of orange peel dried pectin (%)

Response surface methodology (RSM)

A Design Expert (version 6.0.6) software package was used for the experimental design, analyses, and model equation generation for the extraction parameters. Linear, two factorial interaction (2FI), quadratic, and cubic models were used in the analyses and were fitted to the experimental data. The results obtained were compared with the predicted values. Analysis of variance (ANOVA) was performed, and the P value (probability of error value) was utilized in checking the significance of each regression coefficient. The optimal parameters were validated by repeating the experiment at the optimal conditions.

Results and discussion

Effects of processing factors on essential oil yield

The yield of essential oil ranged from 0.57–3.24% (Table 1). This compares favorably with the findings of other researchers on essential oil yield from orange peels. Blanco et al. (1995) obtained maximum essential oil yield of 0.79% for Colombia orange peels using gas chromatography method. Ezejiofor et al. (2011) reported maximum essential oil yield of 0.6% using steam distillation method. Pultrini et al. (2006) achieved maximum essential oil yield of 0.5% using cold pressed extraction method. Sharma and Tripathi (2008) obtained maximum essential oil yield of 1.8% from Indian sweet orange Osbeck epicarp using hydro-distillation method. Hosni et al. (2010) published maximum essential oil yield of 1.89% from Tunisian citrus peels. Bustamante et al.

Table 1 Essential oil yield at various processing conditions

Run	Temperature (C)	Time (min)	EO yield(%)
1	85.00	150.00	1.83
2	100.00	180.00	2.26
3	85.00	210.00	2.84
4	95.00	210.00	3.24
5	90.00	180.00	2.19
6	90.00	180.00	2.04
7	90.00	240.00	2.86
8	90.00	120.00	0.57
9	90.00	180.00	2.17
10	90.00	180.00	2.08
11	90.00	180.00	2.13
12	80.00	180.00	0.62
13	95.00	150.00	2.33

(2016) obtained maximum essential oil yield of 2.3% for Valencia late orange peels using microwave-assisted hydro-distillation method. Giwa et al. (2018) achieved maximum essential oil yields of 4.40, 3.47, and 2.54% using steam distillation, water distillation, and solvent extraction methods, respectively. Even though the results all fall within the range of 0.5 and 5.0% (*w*/*v*) as reported by Palazzolo et al. (2013), the differences could have been due to the nature of the fruits and extraction process vis-à-vis the processing conditions.

In Fig. 1, increase in extraction temperature and time leads to a corresponding increase in essential oil yield. However, at higher extraction temperatures and times, beyond the optimum, essential oil yield decreases. This agrees with the findings of Giwa et al. (2018) who also obtained an increasing trend while working on essential oil extraction from orange peels using water distillation method. At low temperatures, steam travels through the peels slowly and the pressure build up is not sufficient enough to extract the oil out of the peel matrix. However, as the temperature increases for a longer period of time, the oil will eventually break out of the peel matrix and thus be extracted. Nevertheless, extending the extraction duration at higher extraction temperatures caused substantial moisture loss leading to hardening of peels which consequently leads to a decrease in essential oil yield. This supports the findings of Kabuba (2009) while working on steam extraction of essential oils.

Effects of processing factors on pectin yield

The yield of pectin ranged from 12.93–29.05% (Table 2). This compares favorably with the findings of other researchers on pectin yield from orange peels. El-Nawawi and Shehata (1987) and Marin et al. (2007) obtained pectin yield between 21–30 and 13–23%, respectively, while Hashmi et al. (2012), Kanmani et al. (2014), Dehankar et al. (2015), and Khan et al. (2015) reported maximum pectin yields of 20.12, 29.41, 20, and 21%, respectively. These differences could have been due to the nature of the fruits and extraction process vis-à-vis the processing conditions.

It was observed that increase in extraction time with extraction temperature leads to increase in pectin yield, with the maximum obtained at 95 °C and 105 min, beyond which pectin yield levels off (Fig. 2). This agrees with the findings of Pagan et al. (2001), Mollea et al. (2008), and Gama et al. (2015) while working on pectin extraction from peach pomace, cocoa husks, and citric wastes, respectively. As temperature increases, the solubility of the extracted pectin increases which leads to increase extraction rate. However, beyond the optimum temperature, pectin yield decreases because of degradative action which results in pectin of lower molecular size not precipitable with alcohol. Also, it was observed that the longer the extraction duration, the higher the pectin yield. However, at longer durations beyond the optimum, the yield decreases. As the duration increases, the concentration of the pectin in the solution increases, leading to increase yield. However, beyond the optimum time at higher temperatures, thermal degradation occurs which leads to decrease pectin yield. This supports the findings of El-Nawawi and Shehata (1987), Kliemann et al. (2009), Tang et al. (2011), and Gama et al. (2015) while working on pectin extraction from orange, passion fruit, dragon fruit, and citric waste peels, respectively. Increase extraction temperature at low pH leads to corresponding increase in pectin yield (Fig. 3). Low pH signifies increase acid concentration (acidity), and

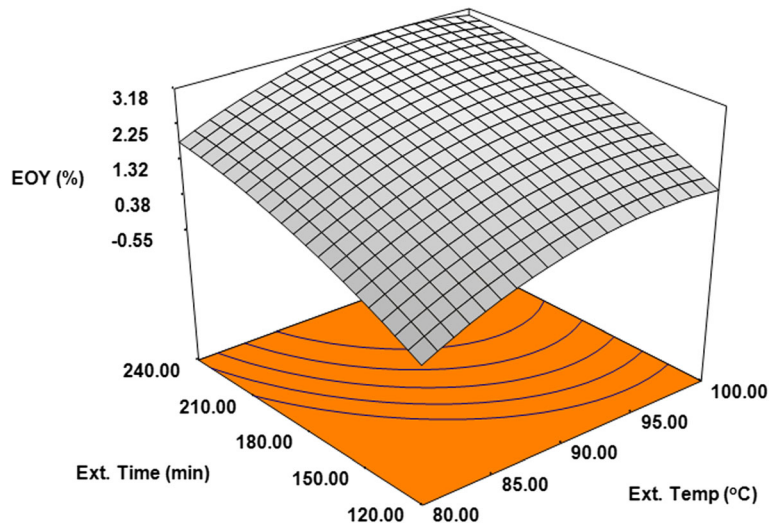

Fig. 1 Response surface plot of the effect of extraction temperature and extraction time on essential oil yield

Table 2 Pectin yield at various processing conditions

Run	Temperature (°C)	Time (min)	pH	Pectin yield (%)
1	90.00	90.00	2.00	19.12
2	90.00	90.00	2.00	19.90
3	95.00	105.00	1.50	29.05
4	90.00	90.00	1.00	27.77
5	85.00	75.00	2.50	14.53
6	80.00	90.00	2.00	13.36
7	90.00	90.00	2.00	18.83
8	90.00	90.00	2.00	18.07
9	90.00	90.00	2.00	18.69
10	95.00	75.00	2.50	14.85
11	85.00	75.00	1.50	15.70
12	90.00	90.00	2.00	19.64
13	90.00	60.00	2.00	13.55
14	85.00	105.00	1.50	24.49
15	90.00	90.00	3.00	12.93
16	95.00	105.00	2.50	23.56
17	85.00	105.00	2.50	22.51
18	95.00	75.00	1.50	16.05
19	100.00	90.00	2.00	26.83
20	90.00	120.00	2.00	25.55

according to Putnik et al. (2017), high acidity level increases extraction yields of various types of pectins and protopectins. This is due to the fractionation of glycosidic bonds in the neutral sugars because they are more sensitive to pH than the link between two galacturonic acids, leading to degradation of the neutral sugar side chains. Pagan and Ibarz (1999) and Pagan et al. (2001) while working on pectin extraction from fresh and stored peach pomace, respectively, observed that at constant extraction time, increase temperature with decrease pH increases pectin yield. The lowest yields of 12.93 and 13.36 were obtained at relatively high pH and low temperature levels, while the highest yields of 29.05 and 27.77 were obtained at relatively high temperature and low pH levels. It was observed that increasing the pH at relatively low temperature leads to drop in pectin yield. This is because high pH level leads to less degradation of the neutral sugar side chains, and at relatively low temperature, solubility of the extracted pectin decreases, hence decrease extraction rate with consequent low pectin yield. Similarly in Fig. 4, increase extraction time at low pH leads to corresponding increase in pectin yield. As observed earlier, low pH signifies increase acidity which increases extraction yields of various types of pectins and protopectins (Putnik et al., 2017). As the extraction proceeds, the pectin concentration in the solution increases. However, at higher durations, the extraction rate gradually decreases because the concentration gradient is reduced and the solution becomes more viscous (Coulson and Richardson 1978). This supports the observation of Maxwell et al. (2012) that increasing the acidity and extraction time increased pectin yield. Also, high concentration of hydrogen ions in the solvent activates the hydrolysis of protopectin at low pH, causing repress of the ionization of the hydrated carboxylate groups by converting them into hydrated carboxylic acid groups (Sereewatthanawut et al. 2008 and Emaga et al. 2008). This loss of carboxylate groups decreases the repulsion of the polysaccharide molecules

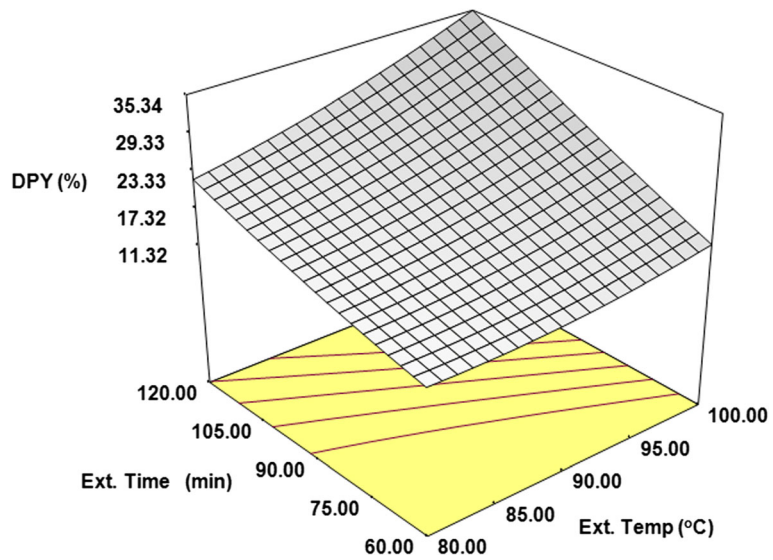

Fig. 2 Response surface plot of the effect of extraction temperature and extraction time on dried pectin yield

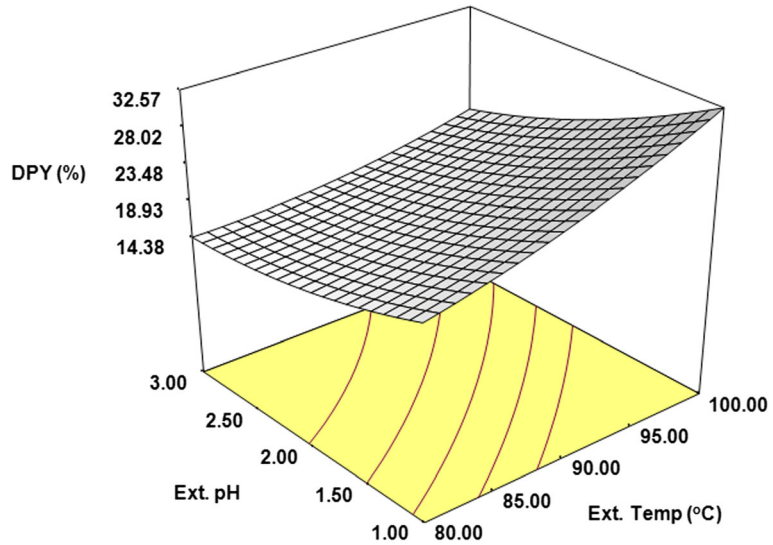

Fig. 3 Response surface plot of the effect of extraction temperature and extraction pH on dried pectin yield

which stimulates the gelation properties of pectin, thereby producing more precipitated pectin at lower pH. As the duration extends, more pectin is produced up to the optimum, after which pectin yield levels off. This trend was established by Maxwell et al. (2012) while studying pectin as an emerging new bioactive food polysaccharide.

Optimization of the oil and pectin extraction yields

When selecting the appropriate model, several considerations were made which are selection of highest order polynomial where the additional terms are significant and there is no error in the model (model is not aliased), insignificant lack-of-fit, and the maximization of the "adjusted R^{2}" and the "predicted R^{2}" (Fakayode and Ajav 2018). However, the cubic model is aliased, which implies that it cannot be selected. For the essential oil extraction, the linear model was suggested (Table 3). The final equation is given as Eq. (15):

$$\text{Essential oil yield}\left(\%\frac{w}{v}\right) = -7.43 + 0.07\text{ET} + 0.02\text{Et} \tag{15}$$

ET = extraction temperature (°C) and E_t = extraction time (min)

There is a direct relationship between the processing factors and essential oil yield as signified by the positive terms in the equation. Both processing factors influence

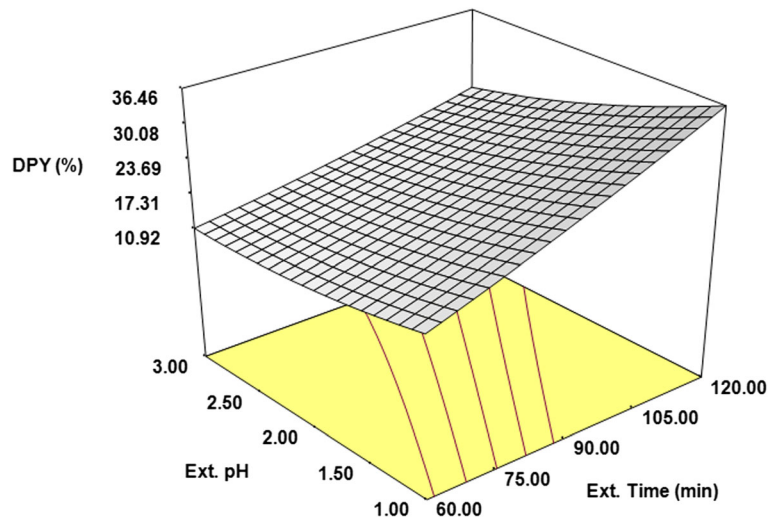

Fig. 4 Response surface plot of the effect of extraction time and extraction pH on dried pectin yield

Table 3 Sequential model sum of squares and ANOVA for response surface linear model for essential oil extraction

Source	Sum of squares	Df	Mean square	*F* value	Prob > *F*	
Mean	56.74	1	56.74	–	–	
Linear	4.98	2	2.49	11.59	0.0025	Suggested
2FI	2.500E−003	1	2.500E−003	0.01	0.9207	
Quadratic	0.62	2	0.31	1.43	0.3006	
Cubic	0.11	2	0.06	0.20	0.8229	Aliased
Residual	1.41	5	0.28	–	–	
Total	63.87	13	4.91	–	–	
Model	4.98	2	2.49	11.59	0.0025	
A	1.46	1	1.46	6.78	0.0263	
B	3.52	1	3.52	16.40	0.0023	
Residual	2.15	10	0.21	–	–	
Lack of fit	2.13	6	0.36	91.80	0.0003	
Pure error	0.015	4	3.870E−003	–	–	
Correlation total	7.12	12	–	–	–	

Values > *0.05* are not significant
A represents extraction temperature, *B* represents extraction time, *df* degree of freedom

essential oil yield (Table 4); however, it was observed that extraction temperature has higher influence on essential oil yield. This agrees with the observation of Giwa et al. (2018) which reported that extraction temperature was the most influential parameter affecting essential oil yield in their research work. The model *F* value of 11.59 implies the model is significant. There is only a 0.25% chance that a "model *F* value" this large could occur due to noise. Values of "Prob > *F*" less than 0.0500 indicate model terms are significant. In this case, *A* and *B* (which represent extraction temperature and extraction time, respectively) are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction will be required for model improvement. However, this was not the case in the chosen model as both model terms are significant. The "lack of fit *F* value" of 91.80 implies the lack of fit is significant. There is only a 0.03% chance that a "lack of fit *F* value" this large could occur due to noise. The "Pred R-Squared" of 0.29 is not as close to the "Adj R-Squared" of 0.64 as might normally be expected. This may indicate a large block effect. "Adeq Precision" measures the signal-to-noise ratio and a ratio greater than 4 is desirable. "Adeq Precision" ratio of 9.73 indicates an adequate signal. This model can be used to navigate the design space. The model was significant with a very low probability value (< 0.0001) and a high coefficient of determination ($R^2 = 0.70$). From Table 4, it was observed that the 2FI model has the same R^2 value as the selected linear model, while the quadratic model (as the cubic model cannot be selected) has higher R^2 value compared to the linear model. However, the linear model has lower standard deviation value compared to the other two. Also, it was observed that for the three models, both

Table 4 Model selection for essential oil and pectin extraction

Essential oil					Pectin			
Model	Linear	2FI	Quadratic	Cubic	Linear	2FI	Quadratic	Cubic
Std. dev.	0.46	0.49	0.47	0.53	2.25	2.37	2.62	0.67
R^2	0.70	0.70	0.79	0.80	0.84	0.85	0.86	0.99
Mean	2.09	2.09	2.09	2.09	19.75	19.75	19.75	19.75
Adjusted R^2	0.64	0.60	0.63	0.53	0.80	0.78	0.73	0.98
C.V.	22.18	23.36	22.31	25.39	11.39	12.01	13.26	3.40
Predicted R^2	0.29	0.01	− 0.50	− 21.70	0.70	0.38	− 0.13	0.79
PRESS	5.05	7.12	10.67	161.73	146.73	304.36	557.50	102.72
Adeq. Precision	9.73	8.00	6.84	5.50	16.80	12.81	9.80	28.08
Significant terms	A, B	A, B	A, B	–	A, B, C	A, B, C	A, B, C	B, C^2, AB, BC, A^3, B^3, C^3

model terms *A* and *B* are significant which shows absolute linearity (Table 4). The high coefficient of determination ($R^2 = 0.70$) showed excellent correlations between the independent variables. This value indicates that the response model can explain 70% of the total variability in the responses.

For the pectin extraction, the linear model was suggested (Table 5). The final equation is given as Eq. (14):

$$\text{Pectin yield (dry matter basis)} = -31.17 + 0.42\text{ET} + 0.26\text{Et} - 4.94\text{pH} \quad (16)$$

ET = extraction temperature (°C), E_t = extraction time (min), and pH = extraction pH

There is a direct relationship between extraction temperature as well as extraction time and pectin yield as signified by the positive terms in the equation, while there is an inverse relationship between extraction pH and pectin yield. All the processing factors influence pectin yield (Table 4); however, it was observed that extraction pH has the highest influence on pectin yield. This was in line with the findings of Pinheiro et al. (2008), Kliemann et al. (2009), Kanmani et al. (2014), and Tiwari et al. (2017) which established that extraction pH was the most significant parameter that influences pectin yield in their research works. The model *F* value of 27.05 implies the model is significant. There is only a 0.01% chance that a "model *F* value" this large could occur due to noise. Values of "Prob > *F*" less than 0.0500 indicate model terms are significant. In this case, *A*, *B*, and *C* (which represent extraction temperature, time, and pH, respectively) are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve the selected model. However, this was not the case in the chosen model as all the model terms are significant. The "lack of fit *F* value" of 16.17 implies the lack of fit is significant. There is only a 0.33% chance that a "lack of fit *F* value" this large could occur due to noise. The "Pred R-Squared" of 0.70 is in reasonable agreement with the "Adj R-Squared" of 0.80. "Adeq Precision" ratio of 16.80 obtained indicates an adequate signal. This implies that the model can be used to navigate the design space. The model was significant with a very low probability value (< 0.0001) and a high coefficient of determination ($R^2 = 0.84$). From Table 4, it was observed that both the 2FI and quadratic models (as the cubic model cannot be selected) have higher R^2 values compared to the linear model, but the latter has lower standard deviation value compared to the other two. Also, it was observed that for the three models, only the model terms *A*, *B*, and *C* are significant, which also shows absolute linearity (Table 5). The high coefficient of determination ($R^2 = 0.84$) showed excellent correlations between the independent variables. This value indicates that the response model can explain 84% of the total variability in the responses.

Validation of selected models for essential oil and pectin extractions

There was an excellent agreement between the observed and predicted values for the essential oil and pectin

Table 5 Sequential model sum of squares and ANOVA for response surface linear model for pectin extraction

Source	Sum of squares	Df	Mean square	*F* value	Prob > *F*	
Mean	7800.46	1	7800.46	–	–	
Linear	410.57	3	136.86	27.05	< 0.0001	Suggested
2FI	7.87	3	2.62	0.47	0.7107	
Quadratic	4.50	3	1.50	0.22	0.8812	
Cubic	65.90	4	16.47	36.63	0.0002	Aliased
Residual	2.70	6	0.45	–	–	
Total	8292.00	20	414.60	–	–	
Model	410.57	3	136.86	27.05	< 0.0001	
A	68.97	1	68.97	13.65	0.0020	
B	243.98	1	243.98	48.21	< 0.0001	
C	97.61	1	97.61	19.29	0.0005	
Residual	80.97	16	5.06	–	–	
Lack of fit	78.75	11	7.16	16.17	< 0.0033	
Pure error	2.21	5	0.44	–	–	
Correlation total	491.54	19	–	–	–	

Values > *0.05* are not significant
A represents extraction temperature, *B* represents extraction time, *C* represents extraction pH, *df* degree of freedom

extractions (Figs. 5 and 6). For the essential oil extraction, in the range of 80–100 °C for extraction temperature and 120–240 min for extraction time where the goal for essential oil yield was maximum, the predicted essential oil yield of 3.38% at extraction temperature of 95.23 °C and extraction time of 231.30 min was obtained with a desirability of 1.00. Under these optimal conditions, the experimental value was 3.34%. For the pectin extraction, in the range of 80–100 °C for extraction temperature, 60–120 min for extraction time, and 1.0–3.0 for extraction pH where the goal for pectin yield was maximum, RSM predicted pectin yield of 30.00% at extraction temperature of 93.07 °C, extraction time of 117.00 min , and extraction pH of 1.60 with a desirability of 1.00. This was experimentally validated as 29.02%. Deviations between experimental and predicted values were low and statistically insignificant for both extractions. This shows that the models chosen can adequately predict essential oil and pectin yields.

Physicochemical properties of essential oil

The physicochemical properties of essential oil are presented in Table 6. The specific gravity of the essential oil was 0.84. The specific gravity determines the weight of the essential oil. It is also important in determining the quality and purity of essential oil. Most of the essential oils have specific gravity ranging from 0.696–1.88 (Pedranti 2011). Specific gravity values of oils are less than 1 for most of the oils except few containing oxygenated aromatic compounds (Osagie et al. 1986). The extracted essential oil has a specific density less than 1 which implies that it is lighter than water and consequently will be insoluble in water.

The free fatty acid of the essential oil was 1.86 mg KOH/g. Rethinam (2003) reported that the maximum value for non-rancid acid is 5.00 mg KOH/g; hence, the extracted essential oil falls within the range of non-rancid acids. Fatty acid composition is a major determinant of oil quality. They are fatty acids which have broken away from oil molecules or triacylglycerols, and their presence indicates that degradation has occurred in the oil. Free fatty acids are less stable and are therefore very prone to oxidation, thereby producing rancidity. It is a key feature linked with the quality and commercial value of oils. The low free fatty acid obtained showed good oil quality.

The acid value of the essential oil was 3.71 mg KOH/g. Essential oils are concentrated and contain several volatile aroma compounds which are majorly free fatty acids. Free fatty acids are considered as degrading in oils because they are responsible for oil rancidity. Oils with low acidity are considered as neutralized and safe for making skin care products as high acidity of oils may be harmful for skin (Kumar 2014). The low acid value of the extracted essential oil indicates that the oil has excellent storage life.

The saponification value of the essential oil was 188 mg KOH/g. Saponification value is an indicator of the average molecular weight and hence chain length. It is inversely proportional to the molecular weight of the oil (Onwuka 2005). High saponification values of oils are due to the predominantly high proportion of shorter carbon chain lengths of fatty acids (Gohari et al. 2011). Low molecular weight (short to medium chain) fatty acids have more glyceride molecules per gram of fat than high molecular weight acids. Each glyceride molecule requires three KOH molecules for saponification; hence, the more the glyceride molecules, the greater the saponification value (Kirk and Sawyer 1991). Nagre et al. (2011)

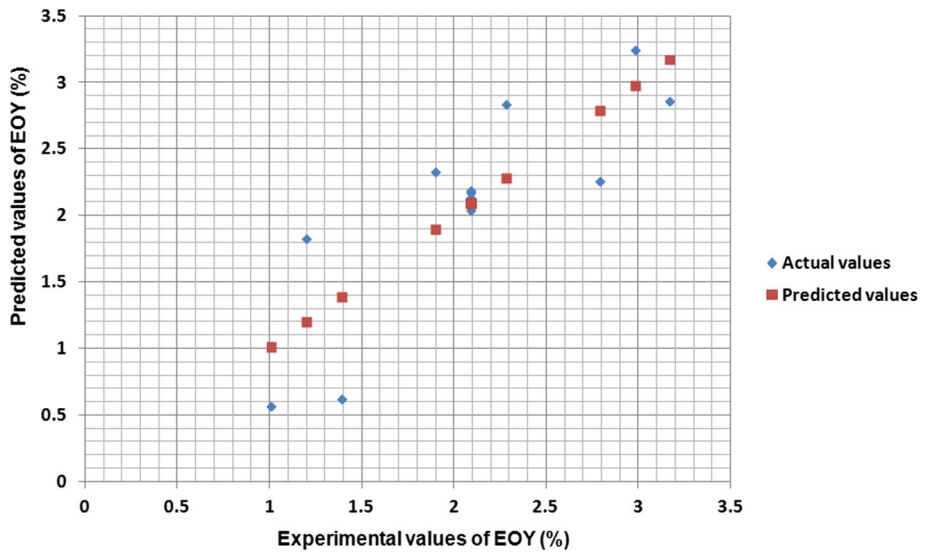

Fig. 5 Predicted and actual values for essential oil yield

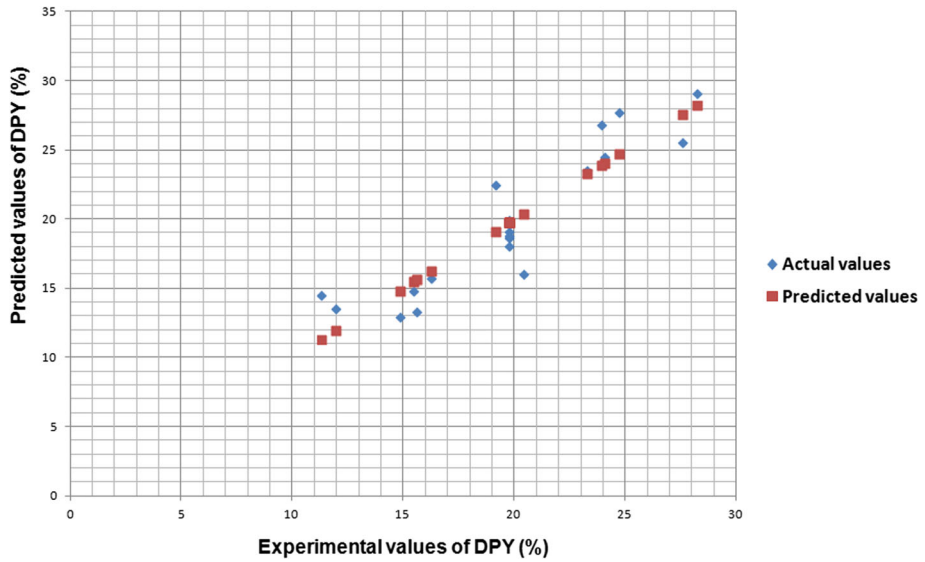

Fig. 6 Predicted and actual values for dried pectin yield

established that saponification value in combination with the acid value gives information on the quantity, type of glycerides, and mean weights of the acids in a given sample. Saponification value is of interest if the oil is for industrial purposes, as it has no nutritional significance (Dari 2009). The larger the saponification number, the better the soap making ability of the oil (Asiedu 1989).

The iodine value of the essential oil was 82 mg I_2/100 mg. The iodine value measures the degree of unsaturation in oil and could be used in quantifying the amount of double bonds present in the oil which reflects the susceptibility of oil to oxidation. It is useful in predicting the drying property of oils, and the moderate value obtained places the oil between non-drying and semi-drying oil; hence, the oil can be used in industries as feedstocks. Low iodine number implies the presence of few unsaturated bonds and hence low susceptibility to oxidative rancidity (Fox and Stachowiak 2007). Therefore, the lower the iodine value, the lower the degree of unsaturation and hence the lower the tendency of the oil to undergo oxidative rancidity.

Table 6 Physicochemical properties of essential oil

Parameters	Values
Specific gravity (at 20 °C))	0.84
Free fatty acid	1.86 mg KOH/g
Acid value	3.71 mg KOH/g
Saponification value	188 mg KOH/g
Iodine value	82 mg I_2/100 mg
Peroxide value	16 mEq O_2/kg
Refractive index (at 20 °C)	1.47

The peroxide value of the essential oil was 16 mEq O_2/kg. Peroxide value gives a measure of the extent to which the oil has undergone primary oxidation. Detection of peroxide shows an initial evidence of rancidity in unsaturated fats and oils. The double bonds found in essential oils play a role in autoxidation (a free radical reaction involving oxygen that leads to deterioration of oils which form off-flavors and off-odors.). Oils with a high degree of unsaturation are most susceptible to autoxidation, and peroxides are intermediates in the autoxidation reaction. Peroxide value is useful for assessing the extent to which spoilage has advanced. The low peroxide value obtained from the extracted essential oil means that the oil will be more stable, and if stored properly, the shelf life will be extended. The International Olive Council (IOC) standard is < 20 mEq O_2/kg oil (Mailer and Beckingham 2006). In general, peroxide levels higher than 20 mEq O_2/kg lead to less stable oil with a shorter shelf life (Mailer and Beckingham 2006). Therefore, the peroxide value obtained from the extracted essential oil shows that the oil is stable.

The refractive index of the essential oil was 1.47. It measures the refraction of light rays as these pass through the oil. The refractive index is a unique number that designates how the oil responds to and bends light. Essentially, it tests how the speed of light is altered when passing through the oil. Kumar (2014) categorized a refractive index of 1.47 as highly pure. Therefore, the refractive index obtained from the extracted essential oil shows that the oil is highly pure. One point worthy of note however is that the refractive index is only a qualitative test of purity of essential oils and does not give percentage purity.

Physicochemical properties of dried pectin

The physicochemical properties of dried pectin are presented in Table 6.

The degree of esterification (DE) of the dried pectin was 60.4%. The degree of esterification is an important molecular index for pectin classification that describes the extent to which carboxyl groups in pectin molecules exist as the methyl ester. The pectin obtained can be categorized as high methoxyl pectin (HMP) because it has a high percentage of degree of esterification greater than 50%. A high degree of esterification allows pectin to form gel quickly at high temperatures, having a more effective action on the lipid profile (Brouns et al. 2012; Dominiak et al. 2014). However, the degree of esterification represents only the ratio between methanol-esterified carboxyl groups and free carboxyl groups, whereas the methoxyl rate refers to the amount of methoxyl groups in a sample (Gnanasambandam and Proctor 1999). Therefore, the degree of esterification should not be assessed separately, as it does not represent the actual amount of methyl esterifications, especially when the galacturonic acid content is low. High methoxyl pectins (HMP) (with DE > 50%) require a relatively high concentration of soluble solids and a low pH for gel formation, while low methoxyl pectins (LMP) (with DE < 50%) form rigid gels by the action of calcium or multivalent cations, which cross-link the galacturonic acid chains (Garna et al. 2004).

The equivalent weight was 599.74. Equivalent weight is a salient physical property of pectin. It is the most important characteristic in determining the functional behavior of pectin. Gelling abilities of individual pectin are tied very closely with equivalent weight. High equivalent weight has higher gel forming effect. Low equivalent weight means higher partial degradation of the pectin which is disadvantageous (Yandav et al. 2017).

The methoxyl content of the dried pectin was 6.23%. Kanmani et al. (2014) established that the methoxyl content of pectin usually varies from 0.2–12% depending on the source and method of extraction. Since the value obtained was below 7%, the dried pectin is of low ester characteristic, which implies that it is desirable in terms of quality. Pectins with low methoxyl content will form a thermo-irreversible gel, which means that it will stay gelled even when heated to temperatures that would normally melt it (Yapo and Koffi 2014). They are used in the food industry to make low-sugar jams because it does not require high-sugar levels to gel and is being utilized for pastries and molecular recipes designed not to be very sweet. They are used as a gelling agent, thickening agent, and stabilizer and can also be used as a fat substitute in baked goods and to stabilize acidic protein drinks such as drinking yogurt (Tiwari et al. 2017).

The total anhydrouronic acid content of the dried pectin was 70.9% which indicates its purity. According to the Food Chemical Codex (FCC), Food and Agriculture Organization (FAO), and European Union (EU), pectin must consist of at least 65% of galacturonic acid (Willats et al. 2006). Anhydrouronic acid content is important to the gelling capabilities of a given pectin. The high value obtained means that the extracted pectin has a low amount of protein.

The acetyl value of the dried pectin was 0.4%. Ranganna (2002) reported that the gelling capacity of pectin decreased with increase in the degree of acetylation. If acetyl group is present in pectin, it inhibits gel formation. The low value obtained makes the pectin to be a good gelling agent.

Conclusions

The effects of processing parameters on essential oil and pectin extractions from orange peels have been established. Essential oil and pectin yields ranged from 0.57–3.24% and 12.93–29.05%, respectively. The predicted optimum value for essential oil yield was 3.38% at extraction temperature of 95.23 °C and extraction time of 231.30 min, while the predicted optimum value for pectin yield was 30.00% at extraction temperature of 93.07 °C, extraction time of 117.00 min, and extraction pH of 1.60. Deviations between experimental and predicted values were low and statistically insignificant. All processing factors have significant effects on both extractions. The models adequately predicted the extraction processes. The suitability of essential oil and pectin for different purposes is determined by their physicochemical properties which is highly important. The physicochemical properties of the essential oil and pectin extracted at the optimum conditions fell within tolerable and acceptable range.

Abbreviations

AC: Ash content of orange peel dried pectin (%); ACV: Acetyl value of orange peel dried pectin (%); AUA: Total anhydrouronic acid content of dried pectin (%); AV: Acid value of orange peel essential oil (mg KOH/g); *B*: Blank titration value (ml); DE: Degree of esterification of orange peel dried pectin (%); DPY: Dried pectin yield (%); EOY: Essential oil yield (%); ET: Extraction temperature (°C); E_t: Extraction time (min); EW: Equivalent weight of orange peel dried pectin (%); FFA: Free fatty acid of orange peel essential oil (mg KOH/g); IV: Iodine value of orange peel essential oil (mg I_2/100 mg); MTC: Methoxyl content of orange peel dried pectin (%); N: Normality of the ethanolic potassium hydroxide (0.1 M); pH: Extraction pH; PV: Peroxide value of orange peel essential oil (mEq O_2/kg); *S*: Sample titration value (ml); SG: Specific gravity of orange peel essential oil; SV: Saponification value of orange peel essential oil (mg KOH/g); *V*: Volume of 0.1 M potassium hydroxide used (ml); V_1: Initial titer (ml); V_2: Final titer (ml); W_a: Weight of ash (g); W_b: Weight of specific gravity bottle filled with oil (g); W_d: Weight of dried pectin obtained (g); W_e: Weight of empty flask (g); W_f: Weight of flask and extracted oil (g); W_o: Weight of oil (g); W_p: Initial weight of orange peel powder used for ex traction (g); W_s: Weight of sample (g); W_w: Weight of specific gravity bottle filled with water (g); *y*: Volume of NaOH from equivalent weight determination (ml); *z*: Volume of NaOH from methoxyl content determination (ml)

Acknowledgements

Authors would like to appreciate the Department of Agricultural and Food Engineering, University of Uyo, Akwa Ibom State, Nigeria, for providing the facilities to carry out the research work.

Availability of data and materials
Research data have been provided in the manuscript.

Authors' contributions
OAF designed the work, analyzed the results, and wrote the paper. KEA carried out the experiments. Both authors read and agreed with the final manuscript preparation.

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Ethics approval and consent to participate
Not applicable

Consent for publication
Not applicable

Competing interests
The authors declare that they have no competing interests.

Publisher's Note

Received: 18 May 2018 Accepted: 7 September 2018
Published online: 23 September 2018

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