Journal of Multidisciplinary Engineering Science and Technology (JMEST)
ISSN: 2458-9403
Vol. 9 Issue 5, May - 2022

Extraction of Pectin from Waste Orange Peels: Influence of Particle Size and Acid Type

Abdullahi S.B. Gimba[1], Abdu Zubairu[2], Abdulhalim M. Abubakar[3], Ayuba Salihu[4] and Petrus Nzerem[5]
[1,4,5]Department of Petroleum and Gas Engineering, Nile University of Nigeria, Abuja Nigeria.
[2,3]Department of Chemical Engineering, University of Maiduguri, Borno State, Nigeria.
Corresponding Author: abduzubairu@yahoo.com

***Abstract*—This work elucidates the effects of particle size and acid type on the yield of pectin extraction from waste orange peels powder and dry cake. The various acids considered in this work are hydrochloric, sulphuric and nitric acid. Three different particle sizes: 0.25nm, 0.15nm and 0.12nm corresponding to standard sieve BSS 60, BSS 100 and BSS 120 respectively were used in this work. The result from shows that the smaller the particle size the higher the yield of pectin extraction for both fresh orange peels powder and dry cake. The effect of the mineral acids shows that sulphuric acid favours highest yield of pectin with 36%, followed by nitric acid (34%) and nitric acid (32%) respectively at particle size of 0.12nm. The solvent extraction, it was found that the smaller the particle size, the higher the volume of oil extracted. Standard sieve BSS 120 i.e. the smallest particle size has the highest yield of pectin (39%) and volume of oil (21ml) for sulphuric acid, which compares favourably with previous works by maintaining a low pH of 1.0. The results were used to develop a predictive model using POLYMATH that correlates the yield of pectin from various acids. Orange peels gives higher yield of pectin in comparison with citrus peel. It is therefore recommended to extract pectin from orange peel waste, while maintaining a small particle size of the dried orange peel at a pH value of 1.0.**

Keywords — pectin, orange peels, extraction, particle size

I. INTRODUCTION

Grape, lemon and orange are all regarded as citrus fruits. Orange is the most widely grown tree plant in the world. A cross-section of *citrus limetta* possess the listed components which includes, oil cells, juice vesicle, outer cuticle layer, stylar or blossom end (distal), stern end (proximal), flavedo, seed and fruit axis. Entirely, an orange is made of juice, seed and peels. Pectin being biopolymer is found in cell walls of many plants in varying amounts [1]. It is a complex mixture of polysaccharides that makes up about one-third of the cell wall dry substance of higher plants [2]. The cell walls of dicotyledonous plants consist of primary and secondary walls. Primary walls, formed in developing cells, are predominantly composed of cellulose, hemicellulose and pectin [3].

Pectin is extracted because of its commercial demand in pharmaceutical, medical, food and cosmetic industry. It is extracted due to its gelling ability in most cases [4]. Figure 1 illustrates world pectin usage by industry. Some of the technologies used in pectin extraction are enzymatic extraction, soxhlet extraction methods, aqueous extraction, microwave assisted extraction, ultrasonic extraction, electromagnetic induction extraction, supercritical fluid extraction methods etc. These techniques involve the use of mineral acids or enzymes.

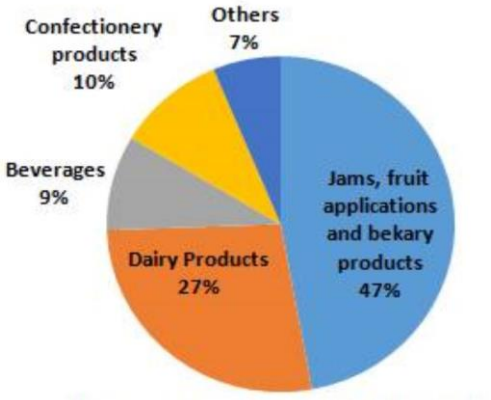

Fig.1.World Pectin Usage by Industry [5]

II. EXTRACTION OF PECTIN

Extraction is the withdrawing of an active agent or a waste substance from a solid or liquid mixture with a liquid solvent. By intensive contact the active agent transfers from the solid or liquid mixture (raffinate) into the solvent (extract). The schematic process flow diagram for the extraction of pectin is presented in Figure 2.

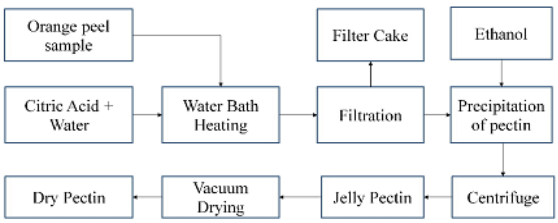

Fig.2. Process Flow Chart for the Extraction of Pectin [6]

Pectin extraction is a multiple stage physical–chemical process in which hydrolysis and extraction of pectin macromolecules from plant tissue and their solubilisation take place under the influence of different factors, mainly temperature, pH and time [7]. Solvent extraction is often preceeded by two-phase separation either by gravity or centrifugal forces.

III. METHODOLOGY

A. Materials

Peels of ripe oranges were collected from Monday market in Maiduguri, Borno State. It was washed to remove impurities. The peels were dried under room temperature at 25^0C for 4 days and kept in a sealed container. Other chemicals and reagents used were of analytical grade obtained from local stores in Maiduguri.

B. Methods

1) Particle Size Analysis

A batch of pieces of dried orange peel were taken and grinded. Three (3) sieve trays were taken. Weight of each empty sieve was measured. A 150g of the grinded material was put in a sieve shaker of the three sieves and run for 10 minutes separately. The sieve trays were arranged on the sieve shaker in order of size, with smallest opening being at the bottom. The trays BSS 60, BSS 100 and BSS 120 were used. Weight of the sieve tray plus sample retained was measured. Weight retained by each sieve tray was calculated. Mesh sizes and weight retained by each tray was tabulated accordingly. The procedure was repeated; a 1000 g of powdered peel was again sieved and weight retained by each sieve calculated. The second sample was used for the extraction of pectin by Soxhlet extraction. The sieving loss is the difference in weight between the original sample and the sum of the recovered fractions, which should not exceed 1% of the original sample weight. If this is greater than 1% then, the sieving process must be repeated.

2) Extraction of Pectin from Dried Orange Peels

A 10g of peel powder with particle size of 0.25nm was measured and put into a 250 ml conical flask. 120 ml distilled water was added. The mixture was stirred vigorously for 10 minutes. The pH of the sample mixture was adjusted to 1.0 by adding mineral acid. The sample mixture was heated at 80^0C for 2 hours in a water bath. Hot acid extract was filtered through a nylon/muslin cloth. Filtrate was allowed to cool at room temperature and its volume measured. A 99.5% (w/w) ethanol was added to the filtered solution and stirred continuously for 10 minutes. This was left for 45 minutes to allow pectin to settle. The coagulated pectin was separated by filtration with filter paper and washed twice with ethanol to further remove any remaining impurity. Pectin was dried using hot air oven. The dried pectin was then weighed. The extracted pectin in crude form was kept in tightly sealed leather.

3) Extraction of Pectin from Dried Cake Residue

The cake residue was obtained by firstly separating the oil from the orange peels powder via aqueous extraction and subsequently extracting pectin from the residual cake residue.

A batch of 70g of orange peel powder was measured from the sieve tray tagged BSS 60. 450 ml of distilled water was added. The mixture was distilled off for 1 hour. The residue was dried to obtain the dry cake. The dry cake was then weighed. The distillate was separated through gravity separation of the two-phase: oil-water mixture. The volume of oil recovered was measured.

The procedure adopted for the extraction of pectin from fresh grounded orange peel described in previous section was repeated for the extraction of pectin from the dry cake residue.

4) Pectin Yield

The yield of pectin obtained was determined using modified method of Seggiani et. al. (2009) [8].

$$Yield(\%) = \frac{Pure\ pectin\ (g)}{Initial\ dry\ orange\ peel\ (g)} \times 100\% \qquad (1)$$

where the word “pure pectin” stands for the pectin obtained on moisture and ash free basis.

Equation (1) can be modelled using POLYMATH application to give the yield of pectin based on inputting parameters that directly affect the quantity of pectin obtained. From the results that was obtained, these parameters were determined by carefully analyzing them, and the parameters were correlated using multiple linear regression. Several model equations were developed corresponding to the various acid types used in this work.

IV. RESULTS AND DISCUSSION

A. Particle Size Distribution

The results of the sieve analysis with 150g and 100g of sample were presented in Table I and II respectively. Sieving loss was calculated and shown in Table III. It could be seen that for both sample batches, the sieve loss was less than 1.0% which agrees well with literature recommendations.

TABLE I. SIEVE ANALYSIS OF ORANGE POWDER OF 150G BATCH

Sieve Tag	Particle Size(nm)	Weight of Sample Retained (g)
BSS 60	0.25	71.4
BSS 100	0.15	43.0
BSS 120	0.12	34.6

TABLE II. SIEVE ANALYSIS OF ORANGE POWDER OF 1000G BATCH

Sieve Tag	Particle Size(nm)	Weight of Sample Retained (g)
BSS 60	0.25	476.0
BSS 100	0.15	290.0
BSS 120	0.12	232.0

TABLE III. SIEVING LOSS OF ORANGE POWDER

Weight of Sample (g)	Sum of Weight Retained (g)	Sieving Loss (%)
150	149	0.67
1000	998	0.20

It is evident from Table I and II that as the particle size increases the weight retained also increases regardless of the batch size of sample. The quality of the sieved sample also depends on the sieving time which not investigated in this work.

B. Pectin Yield from Orange Peels Powder

The yield of pectin as function of particle size for various acid types is presented in Figure 3. The yield of pectin decreases with increase in particle size for all the acid types used. This is attributed to the decrease in surface area available for extraction as particle size increases. Overall, sulphuric acid together with particle size 0.12 nm (BSS 120) performed better in terms of yield of pectin (36%), followed by nitric acid (34%) and hydrochloric acid (32%). Generally, it can be deduced that, the smaller the particle size, the higher the yield of pectin.

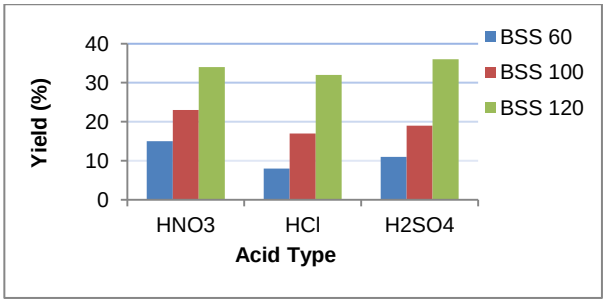

Fig.3. Pectin Yield as Function of Particle Size for Various Acid Types

C. Pectin Yield from Dried Cake Residue

The yield of pectin from the dry cake residue is as shown in Figure 4. Considering the the smallest particle size tagged BSS 120 has a highest yield of pectin for all acid type. It was 37% for nitric acid, 35% for hydrochloric and 39% for sulphuric acid. This was followed by BSS 100 where nitric acid gave the highest yield of 27%. The largest partcle size (BSS 60) indicated nitric acid with the highest pectin yield (19%). Thus, it can be deduced that the smaller the particle size of sample of grounded dried cake obtained after oil extraction, the higher the yield. From the foregone, sulphuric acid has the highest yield of pectin from followed by nitric acid and hydrochloric acid accordingly. Nonetheless, the difference in theyield of pectin is not considerably significant among the acid types.

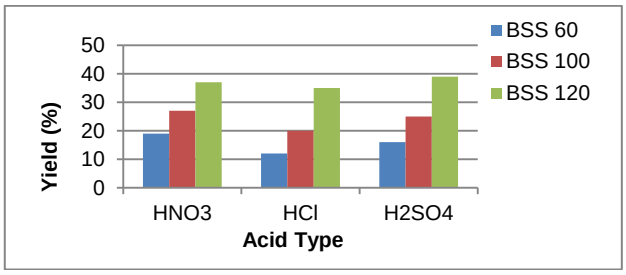

Fig.4. Pectin Yield as Function of Particle Size for Various Acid Types

Generally, particle size of sample has proven to be an important factor in pectin extraction from citrus (orange) peel. The smallest particle of 0.12 nm produces highest pectin yield (36%) via the extraction from fresh orange peel powder, and 39% pectin yield from dried cake. The results also indicated that for the acid types, sulphuric acid has the best yield of pectin for both two techniques. It also emphasizes the significance of separating the oil from the orange peels powder before extracting pectin from dried cake, which manifest in the slight increase in the amount of pectin recovery.

D. Regressional Model of Pectin Yield

It has been identified that filtrate and particle size are the dominant factors that influence the amount of pectin yield during either direct extraction from dried orange peel powder or extraction from dried cake residue after oil separation. These two parameters were correlated with pectin yield using POLYMATH data regression tool. The resulting polynomials for the various acid types used are presented in Table IV.

Acid	Model Equation
HNO_3	$Y_N = 0.5905428F - 16.9257S$
HCl	$Y_H = 0.9683831F - 100.3073S$
H_2SO_4	$Y_S = 0.5031071F - 37.36623S$

TABLE IV. REGRESSION MODELS FOR VARIOUS ACIDS

Where: *F* is the filtrate volume (ml) and *S* is the particle size (nm)

The resulting polynomials were validated through data fitting and the predictive estimate using the equations for the various acid types are shown by comparison with the experimental values as presented in Figure 5 - 7.

It could be seen that the polynomials prediction was only about 18% maximum deviation for all acid types considered.

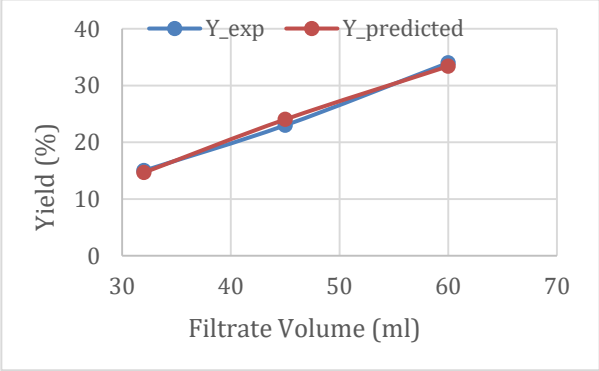

Fig.5. Pectin Yield as Function of Filtrate Volume for Nitric Acid.

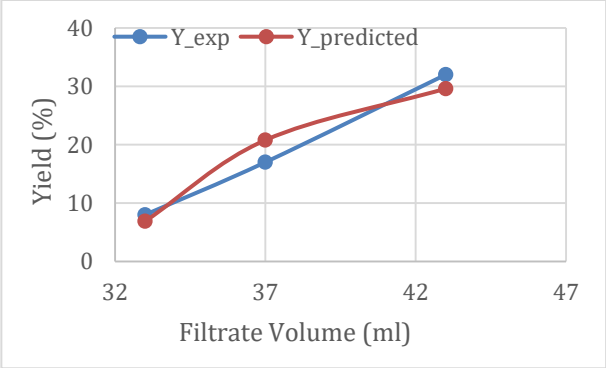

Fig.6. Pectin Yield as Function of Filtrate Volume for Hydrochloric Acid

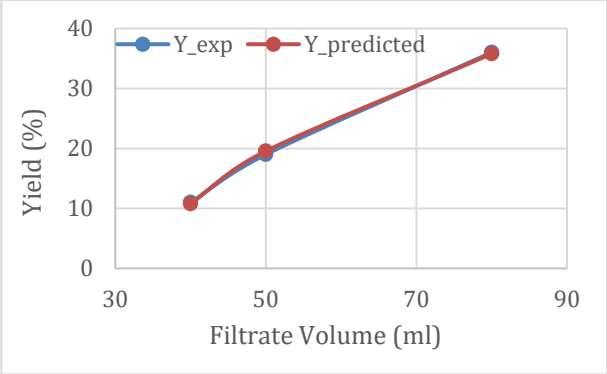

Fig.7. Pectin Yield as Function of Filtrate Volume for Sulphoric Acid

V. CONCLUSION

From the foregone results, we conclude that regardless of the kind of acid employed in the extraction of pectin from orange peels, the yield of pectin increases with decreasing particle size. There is only slight increase in pectin recovery from dried orange peels cake that was stripped of the oil, compared to pectin yield from fresh dried orange peels powder. The effect of acids on pectin yield shows that f pectin recovery decreases in the order of H_2SO_4 , HNO_3 and HCl respectively for all particle sizes as well as for both fresh orange peel powder and the dried cake samples. Pectin yield from both orange peel powder and dry orange peel cake was successfully correlated with the particle size and amount of filtrate by simple polynomial to generate predictive model relating yield, filtrate volume and particle sizes developed using POLYMATH Regression Tool.

REFERENCES

[1] A. Chakraborty and R. Subhajit, “Development of a Process for the Extraction of Pectin from Citrus Fruit Wastes viz. Lime peel, Spent guava extract, Apple pomace etc”. *Internet Journal of Food Safety* , vol. *13*, pp. 391-397, 2017.

[2] P. Sriamornsak, Chemistry of Pectin and Its Pharmaceutical Uses: A Review, pp. 206-222, 2002.

[3] P. Harris and B. A. Stone, “Chemistry and Molecular Organization ofPplant Cell Walls, *Biomass Recalcitrance*, pp. 60-93, 2008.

[4] K. A. Castillo-Israel, S. F. Baguio, M. D. Diasanta, R. C. Lizardo, E. I. Dizon and M. I. Mejico, “Extraction and characterization of pectin from Saba banana [Musa ‘saba’(Musa acuminata x Musa balbisiana)] peel wastes: A preliminary study”. *International Food Research Journal* , vol. 22 (1), pp. 202-207, 2015.

[5] G. N. Kanse, S. Chirag, S. Swapnil and S. Vishal, “Extraction of Pectin from Orange Peel’s and Its Applications: Review”, *International Journal of Innovative Research in Science, Engineering and Technology (IJIRSET)* , vol.*6* (9), pp. , 2017.

[6] S. Pandharipande and H. Makode, “Separation of oil and pectin from orange peel and study of effect of pH of extracting medium on the yield of pectin”, *Journal of Engineering Research and Studies*, pp. 6-9, 2012.

[7] Z. I. Kertesz, *The pectic substances.* New York: Interscience Publishers, pp. 1951.

[8] M. Seggiani, M. Puccini, M. Pierini, S. Giovando and C. Forneris, “Effect of different extraction and precipitation methods on yield and quality pectin”, International Journal of Food Science and Technology, vol. 44 (4), pp. 574–580, 2009.