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Studying the Effect of Extraction Parameters on Extracting Process of Pectin from Dried Orange Peels

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ABSTRACT

The most commercial source of pectin are orange peels. This paper explains extraction Pectin from orange peels, juice processing industry waste using simple method with pectin yield 15.25%. The main objective of the present work is to prove the importance of using raw material of food processing industries such sweet orange peels in produce a useful product like pectin. Experimental observations affirm that the extraction of pectin from peels give high yield than filtration residue when taken after extracting orange oil through distillation. The most appropriate method for industrial production of pectin is the process by which orange oil is first extracted using the simple distillation technique followed by the extraction of pectin. The potential benefits of industrial pectin extraction from an economic and environmental point of view is extraction of pectin from orange peels process.

1. Introduction

Pectin is present in many plants and it is used in the food industry as a gelling agent. Pectin can be used as liquid extract or as dried powder, but the dried powder is easier than a liquid in storing and handling. Pectin used in pharmaceutical, biotechnology, food and beverage industry [1], thickening agent, gelling agent and colloidal stabilizer. Pectin is no longer just a gelling agent but also used in wide applications as fruit products for the food industry, dairy products, desserts, soft drinks and pharmaceuticals'.

The most source of pectin is citrus peel and apple pomace [2]. Mostly Citrus peel has often been preferred material for pectin production due to its high pectin content and good color properties. Generally lemon and lime peel are the preferred sources of citrus pectin [3]. The amounts of pectin from these different sources varies considerably as in Apple pomace (10-15%), Citrus peel (25-35%) and Sugar beet (10-20%).

The most common type of pectin is the sweet orange peel [4]. That extracts pectin from the sweet orange peel which is a waste for the orange juice processing industry. The best way to produce industrial pectin is to extract pectin using a bathing or drying technique. Extraction of pectin from peel when taken after extracting orange oil through simple distillation gives higher yield than leaching residue [5]. The process in which orange oil is first extracted using technique of simple distillation followed by acid extraction of pectin is most suitable for industrial production for isolation of pectin. Pectin extraction in a hot diluted strong mineral acid solution is the most commonly used method [6].

In this paper different parameters studied and its effect on pectin yield, the size of the crushed orange peels, extraction time, and the pH values of acidic solution used for the extraction of pectin. The orange

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peels are sun dried till their moisture content is negligible. Then crushed to 16, 32 and 60 mesh screens which used to separate the powdered peels accordingly. 80gm of 16 mesh size peel powdered is taken and fed to the Soxhlet apparatus with 1000mL of petroleum ether (taken as solvent). The setup is maintained at 40^{O}C (B.P. of Petroleum ether) for 6 hours, 9 extra hours and 12 hours respectively. At bottom of the solvent the oil collecting then separated by simple distillation, then powdered peels are collected separately after the solvent extraction is complete.

The final stage done is prepared a citric acid solution of pH 1.0, 1.5, 2.0 and 2.5 which heated for 30 minutes with 5gms of powdered peels at temperature 65^{O}C with continuous stirring. After cooling the solution, it filtered. The filtrate was added to double amount of ethanol and allowed to precipitate. Pectin was subsequently washed with ethanol two times, then it dried in a hot air oven at 400C for 20 min. The process was repeated for 32 and 60 mesh size.

2. Extraction of Pectin from Orange Peels

2.1. Materials and sample preparation

After juice extraction from the oranges, the peels were washed with running water for 10 minutes to remove seeds and sugar residuals. The peel was then drained and pressed for removing water excess thereafter it was shredded into 1cm diameter pieces, before it was dried in a vacuum dryer. The drying temperature was variable for different trials and peels were weighed before drying and then after each 1 hour of drying. To estimate the time required to reach a moisture content of 8-10% at each drying temperature.

The moisture content should be kept between 8-10% because if it becomes less than 8% the pectin chains will undergo degradation and if it exceeds 10% this will affect the dry powder shelf life issue taking this to a profitable industry.

Orange fruits (10 pieces) was used. The fruits washed clearly by water after remove any foreign materials. Total pectin was determined as g/100 g on fresh weight basis sample. Orange were peeled and dried for 4 days then powdered. Orange powder (500 g) were used. Then 2.5liter distilled water and 25 ml HCL were added for each blend and then mixed and left for 24 hours, then filtered in separation device. 0.5liter of filtration results added to 0.5liter ethanol (95%), the mixture was put into centrifugation apparatus. Then left one hour and filtered through Buchner funnel. Acidified ethanol adds to residues. The filtrate was washed with 125 ml acetone for drying and filtrate was dried at normal room temperature for 24 hours. The product breached into fine powder and sieved by 40 mesh sieves to separate pectin from fiber. The pectin powder collected, weighed and then packed in plastic container pending jam production.

2.2. Drying

The drying trials were held in a Sci finetech vacuum dryer in the Egyptian National Research Centre facility department of chemical engineering, and the choice of using a vacuum oven was to get higher effect for low temperature as pectin is very temperature sensitive and it starts degradation at 80oC and also for reducing drying time which could have taken days in a conventional dryer. Drying of the fresh peels is performed at different temperatures (60, 70, and 80oC) to check drying time required to reach 8-10% of moisture for optimize the processing conditions. And to check if drying temperature has a significant effect on yield and quality of the extracted pectin. At 60oC the sample took around 7 hours to reach constant weight (0% moisture), and looking to the curve we find that we will at achieve 10% moisture after about 4 hours of drying at 60oC so another sample papered and dried it for 4 hours at this temperature for extraction as appeared in Figure1

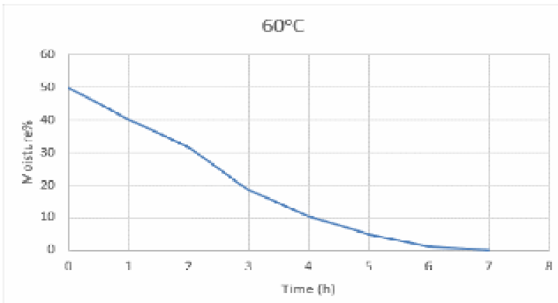

Fig. 1: Decrease the Moisture % with Time at 60^{O}C

The same weight of shredded orange peels is dried at 70^{O}C this time and it took around 6 hours and 40 minutes to reach constant weight and it's found from the curve that 10% moisture is achieved after nearly 3 hours and 10 minutes. So, a dried sample prepared at this temperature for 3 hours and 12 minutes for pectin extraction as in Figure 2.

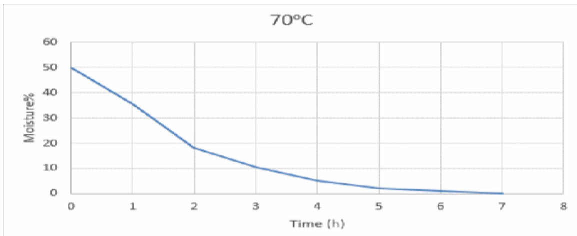

Fig. 2: Decrease the Moisture % with Time at70^{O}C

The next trial was at 80 ^{O}C and the same weight of peels took 5 hours to reach zero moisture and 2 hours and 20 minutes to reach 10% moisture as in Figure 3, which is will be the working condition.

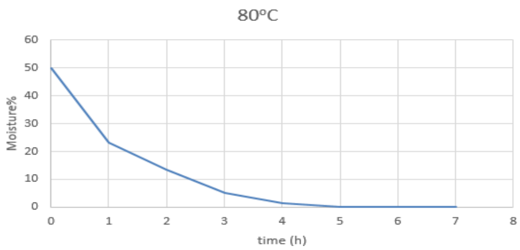

Fig. 3: Decrease the Moisture % with Time at 80^{O}C

2.3. Extraction Process

Figure 4 explain the extraction and isolation of pectin from fresh orange peel sample.

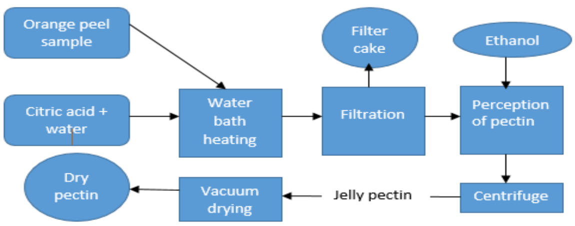

Fig. 4: Steps of Extraction Pectin from Orange Peel Sample

Extraction of pectin affected by many parameters such as PH, temperature, solvent used for extraction, time of extraction. The detail parameters are discussed below.

2.3.1. pH

pH (scale used to specify how acidic or basic a water-based solution) is one of the more crucial parameters affecting the amount and properties of extracted pectin. Pectin yield decreased with increasing the pH value and vice-versa.

2.3.2. Temperature

Yield of pectin decrease in lower temperature while at high temperature it Combustible.

2.3.3. Solvent used for extraction

Citric Acid (C6H8O7), Hydrochloric Acid (HCL), Sulphuric Acid (H2SO4), Nitric Acid (HNO3), EDTA, Ammonium Oxalate (C2H8N2O4) and oxalic acid (C2H2O4) used in extraction process but the high yield is obtained through Citric Acid as a solvent. The yield of pectin extraction is reported in literature up to 55-60% by using Citric Acid as a solvent.

2.3.4. Time of extraction

Pectin yield increased with increasing the time of extraction and decreased from the maximum level due to the thermal degradation of the extracted pectin.

2.3.5. Agitation Rate

Yield of pectin increase with increase of agitation rate as a result of increasing stirring rate and reduce the thickness of the diffusion layer which can enhanced the extraction process.

2.3.6. Liquid Solid Ratio (LSR)

Pectin yield increased firstly and gradually decline with increasing LSR is reported in literature. for increasing of dissolving capacity when LSR is increased but reduced the pectin separation from the solution. As for the other reason, when the LSR increase from certain values degradation of pectin increases with the decrease of pectin concentration in the solution. Related to low content of raw material that provided less protection for the dissolved pectin and facilitated to the degradation of pectin. Citric acid in distilled water solutions of desired pH values 1, 1.5, 2, 3, 4 and 5 prepared. Orange peel samples weighing 10 gm each are dipped in to the solution and heated at 80C for 10 minutes. The solution cooling, then filtered using cloth filter and filter paper under vacuum. Ethanol is added to the filtered solution for facilitating filtration of pectin. The solution is filtered through centrifuge at 8000 rpm for 15min at 100 C to separate jelly pectin which is dried under vacuum at 50 c and 100mmHg gauge for two hours. Dried pectin is thus obtained as given in Table 1.

Table 1. Different Values of pH

Solution of PH	pH 1	pH 1.5	pH 2	pH 3	pH 4	pH 5
Volume taken for extraction (g)	100	100	100	100	100	100
Amount of peel (g)	10	10	10	10	10	10
Extraction temperature (oC)	80	80	80	80	80	80
Extraction time (minute)	5	5	5	5	5	5
Volume after filtration (ml)	89	50	70	73	88	70
Volume of ethanol (ml)	44.5	25	35	37	44	35
Centrifuge (rpm)	8000	8000	8000	8000	8000	8000
Centrifuge time (minutes)	15	15	15	15	15	15
Weight of dried pectin (g)	4.55	2.21	1.15	0.28	-	-
% yield of pectin	45.5	22	11.5	2.8	-	-

3. Yield

pH is one of the more crucial parameters affecting the amount and properties of extracted pectin. Figure 5 shows that pectin yield decreased with increasing pH, highest being 52.90% at pH 1 and 60-mesh size. Presence of high concentration of hydrogen ions in the solvent has stimulated the hydrolysis of protopectin [7]. When the PH decreases, the concentration of hydrogen increases in the solution. The ionization of the carboxylate groups is suppressed, i.e., the highly hydrated carboxylate group is converted into hydrated carboxylic acid groups [8]. The loss of carboxylate groups can reduce the antagonism of the polysaccharide molecules that enhance the properties of pectin gelation properties giving more pectin participation at lower PH. This observation agreed with previous work, which extracted pectin from apple pomace and sugar beet pulp where the yield increased with increasing acid strength. Further, increase in the yield of pectin is also noted as the size of the powdered peels decreased [9]. This happens as the surface area available for mass transfer increases with the decreasing size thus increasing the yield. Pectin yield (%) = [(Mass of dry pectin (g)/ Mass of orange peel sample (g)) *100]

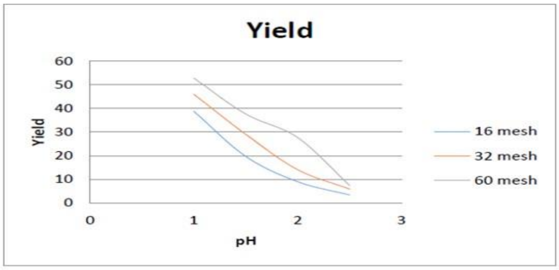

Fig.5: Variation of Yield of Pectin Extracted from 16, 32 and 60 Mesh Size Peels with Respect to pH

4. Results and discussion

Different variables where assessed regarding their influence on pectin yield from dried orange peel powder.

4.1. Effect of pH

Extracted pectin yield is function of the concentration of hydrochloric acid (represented as acid pH) as in Figure 6. The lower acid concentrations were not enough to hydrolyze the insoluble pectin constituents. The optimum PH value was 1.7. The values of pH is selected from literature [10]

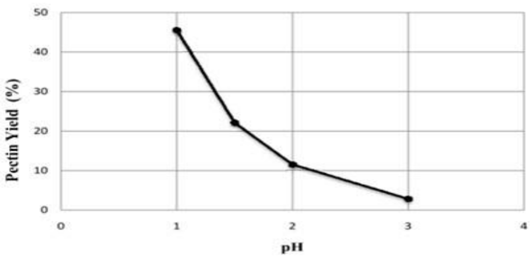

Fig. 6: pectin Yield (%) vs. pH

4.2. Effect of Extraction Temperature

As in Figure 7, the temperature had a marked influence on the extraction of pectin. The yield was optimum at about 90°C. Temperature increases in the range from 50-90°C the solubility of the extracted pectin will increase, giving a higher rate of extraction; further the diffusion coefficient will be expected to increase, and this will also improve the rate. A low temperature is not enough for the hydrolysis of protopectin (the insoluble form of pectin). The decrease of the yield obtained by increasing the temperature above 90°C is due to degradative action resulting in pectin of lower molecular size not perceptible with alcohol.

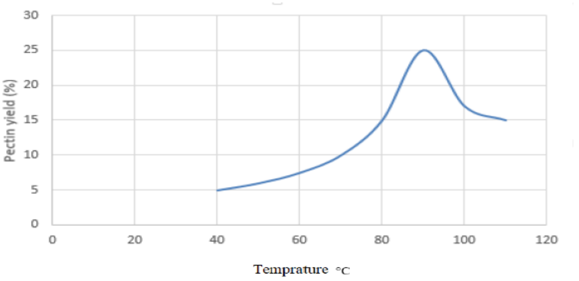

Fig. 7: Pectin Yield (%) vs. Temperature (°C)

4.3. Effect of Extraction Time

As the extraction progresses, the concentration of the pectin will increase in the solution and the extraction rate will gradually decrease; first, because the concentration gradient will be reduced and, secondly, because the solution becomes more viscous. Relatively long extraction period (t > 90min) may cause a thermal degradation effect on the extracted pectin, thus causing a decrease in the amount perceptible by alcohol.

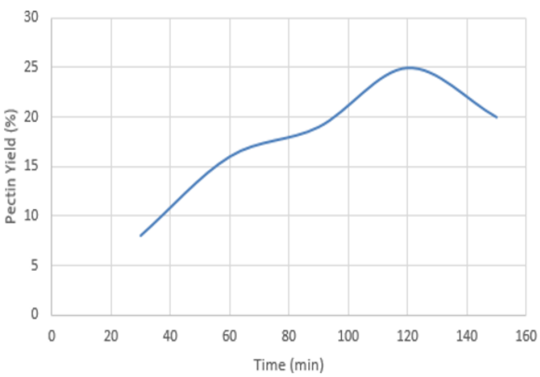

Fig. 8: Pectin Yield (%) vs. Extraction Time (min)

4.4. Effect of Agitation Speed

The effect of agitation of the solvent is important as it increases the eddy diffusion and therefore increases the transfer of material from the particles surface to the bulk of the solution. Agitation also prevents sedimentation and is more efficiently used is for the interfacial surface. Once the solids are completely suspended further expenditure of agitator power does not produce commensurate improvement in the mass transfer rates.

Agitation is also important in the case of solutions containing fibrous solids, to overcome the relatively increasing viscosity of the extraction mixture, since large changes in diffusivities usually accompany corresponding changes in viscosity. For relatively dilute solid -liquid mixtures, except for fibrous solids, the power to agitate at a given speed is essentially the same as for the clear liquid. Slurries containing fibrous solids are likely to be non-Newtonian in character, i.e. the velocity gradient in the moving mass is not directly proportional to shear stress. The viscosity of such fluids is related to agitator power.

The data of Figure 9 show that there is no significant increase in the yield of the extracted pectin with increasing the velocity of agitation, N, of the extraction mixture. This may be because the resistance of mass transfer of the extracted pectin from the peel surface to the bulk of the solvent is quite small compared to the resistance to diffusion within the peel itself. It can be considered that the resistance to mass transfer within the solid is likely to be the controlling resistance and that of the liquid surrounding the solid to be quite minor. Therefore, the degree of agitation of the external solvent has no appreciable effect on the extraction rate.

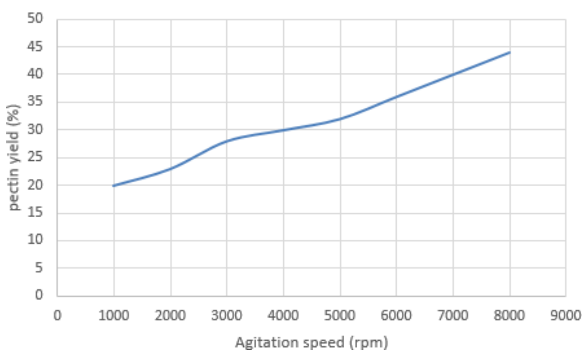

Fig. 9: Pectin Yield (%) vs. Agitation Speed (rpm)

5. Conclusion

The present work is dedicated for the development of the part of the process technology needed for the extraction of value-added products, pectin from orange peel, which is the waste of orange juice processing industry. The results of the experimentally work appeared that;

- Sweet orange peels are good source of pectin and have the potential to become important raw material for food processing industries.
- The source of the peel, to extract the pectin, when taken after extracting orange oil by a simple distillation yields higher yields than leaching residue. Therefore, we can conclude that the process in which the extraction of oil for the first time using simple distillation technique followed by the extraction of pectin using acid is most suitable for industrial production.
- Extraction of pectin successfully provides the potential benefits for industrial extraction of pectin from an economic and environmental point of view.
- Pectin extracted from orange peels 31.5g which equivalent to 15.25% of total orange peel weight.
- Pectin yield increase as the pH decrease in pH range from 1 to 3, the optimum pH value is 1.7.
- Pectin yield increase as the temperature increase till 90 ºC after that it decreases.
- Pectin yield increase as agitation speed increase in the range of (1000 to 8000 rpm)

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