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YIELD OPTIMIZATION AND CHARACTERIZATIONS OF EXTRACTED PECTIN FROM CITRUS FRUITS SOLID WASTE UNDER MICROWAVE ILLUMINATION

[1]Dipak D. Dhobi, [2]Hemangi Desai

[1]Government Science College, Bhilad, V.N.S.G.U., Surat 396105 Gujarat (INDIA) dhobidipakd@gmail.com
[2]Shree RamKrishna Institute of Computer Education and Applied Science,
Sarvajanik University., Surat 395001, Gujarat (INDIA)
hemangal4@yahoo.co.in
Tel: +919724915932

Abstract: Citrus fruits like oranges, lemons, grapes, peaches, strawberries and apples deserve special attention for remarkable production of pectin. In the present work we have extracted pectin first time from these citrus fruits solid waste obtained from greengrocery dustbin. An optimization study for extraction of pectin from these citrus fruit solid waste using variables like pH, microwave illumination time (sec.) and illumination power (W) was performed and optimal conditions (1.5 pH, 120 s illumination time, 300 W illumination power) were resoluted with maximum (34.55%) pectin yield. The structure of extracted product was confirmed through structural characterizations like FT-IR and elemental analysis.

***Index Terms*:** Citrus fruits solid waste; Pectin extraction; Microwave illumination.

I. INTRODUCTION

Packed drinks and fresh juice are most probably made up of citrus fruits. Roughly half of the fruits' mass is made up of citrus peels and are the vast by-product of fruits industrial processing [1] [2] [3] [4]. These citrus fruits waste are the rich source of biodegradable polymer - pectin. Sugar beet pulp, strawberries, peaches, grapes, lemon, apple and orange are main sources of pectin from which apple pomace and orange peel contain high levels of pectin [5] [6] [7] [8] [9].

Pectin is methoxylated galacturonic acid residues contained daedal heteropolysaccharide based on 1, 4-linked α-d-galacturonic acid structure which is widely spreaded in nearly all the vegetables and fruits as the structural unit of cells [10] [11] [12]. Its applications embrace ice cream, gelling agent, emulsified meat products, thickener, salad dressings and stabilizer in variety of cosmetic, pharmaceutical and fruits products [13].

In excess of about 30,000 tons pectin is demanded by the world market [5]. So, it is important to develop new techniques to extract pectin in a shorter time with excellent quantity and quality. Less solvent consumption, shorter time, lowest cost and higher extraction rate with better yield are the advantages of microwave assisted extraction (MAE). Extraction through Soxhlet technique and supercritical fluid extraction (SFE) generally takes number of hours, whereas MAE which is eco friendly, cheaper, simpler and requires only a few minutes [14], increases cellular characteristics and thus the water absorption potential and improves the yield [15]. Pectin has wide applications in the food industries. Since, the modification of citrus waste into precious outcome like pectin contrive interstice for the reduction of the environmental pollution. To give the new direction to pectin extraction with maximum yield from citrus waste using MAE is the main purpose of this study. The other pectin extraction methods include direct boiling of reaction content [17] [18] [19]. Conventional acid extraction method using acidic hot water under specific experimental conditions caused degradation and objectionable changes in functional and physicochemical properties due to direct heating for long time [7]. Currently, to obtain increased quantity and quality of extracted pectin from various resources, ultrasonic treatment and super high electromagnetic frequencies are employed. [20] [21] [22] [23] [24]. In the food industry Ultra-high pressure (UHP) is applied pertinent to enzyme inactivation, food preservation, micro organisms avulsion and biopolymer altered conformation [25] [26] [27]. UHP method cause structural changes like protein denaturation and cell deformation [26] [28] [29]. Since, the solubility increases with pressure [30], this method was employed for the extraction of bioactive constituents from natural products [31] [32] [33].

In order to maximize yield and improve the quality of extracted pectin the execution of ample method is momentous. For the cogency of extraction conditions on operative properties and physicochemical characteristics of the extracted pectin yield from different natural sources the numerous research reports have been studied. [7] [17] [34] [35]. The previous pectin study demonstrated that beside intrinsic factors like average molecular mass, degree of methyl and acetyl esterification, galacturonic acid and sugar content, but also extrinsic factors like ionic strength of solution, ash content and pH value also affect its operative properties [6] [7] [36] [37] [38].

To discover the expedient method for the pectin extraction from citrus waste and to optimize MAE condition for better pectin product is the aim of this study.

II. EXPERIMENTAL

2.1. Raw materials and reagents

Citrus fruits (oranges, lemons, grapes, peaches, strawberries and apples) waste obtained from local greengrocery near Silvassa, Dadra and Nagar Haveli, India was soaked in water bath for fifteen minutes at 85 ^{0}C temperature for its enzyme inactivation and dried for 11 hours under sunlight which is under cutting and drying at 70 ^{0}C to get constant weight. After then to get its fine powder form it was milled in a Nutribullet kitchen blender and stored in dry area within plastic bag former to the experiment. All chemicals utilized in this work were laboratory grade bought from Merk chemicals, Mumbai, India.

2.2 Pectin extraction with microwave assistance from citrus fruits solid waste finely ground flour

As shown in Fig. 1 the indoor microwave oven (SAMSUNG, SHM9187W) having 2450 MHz operating frequency with adjustable time and power was used for pectin extraction. In Fig. 1 the experimental design for pectin extraction from citrus fruits solid waste fine powder is depicted. 1 g weight of citrus fruits waste fine flour dissolved in 30 ml distilled water (solid-liquid ratio 1:30) setting different pH values 1.0, 1.5 and 2.0 using hydrochloric acid 0.1, 0.032 and 0.001 mol/l respectively and transferred to 250 ml Borosil beakers which were then subjected to rotating disc of microwave at different illumination power (180-300-450W) for the specific time (60-120-180 s) followed by cooling down of these Borosil beakers at room temperature and filtered with Whatman paper no-2. After skipping residue the filtrate was centrifuged and obtained supernant was precipitated with an equal amount of 95% ethanol.

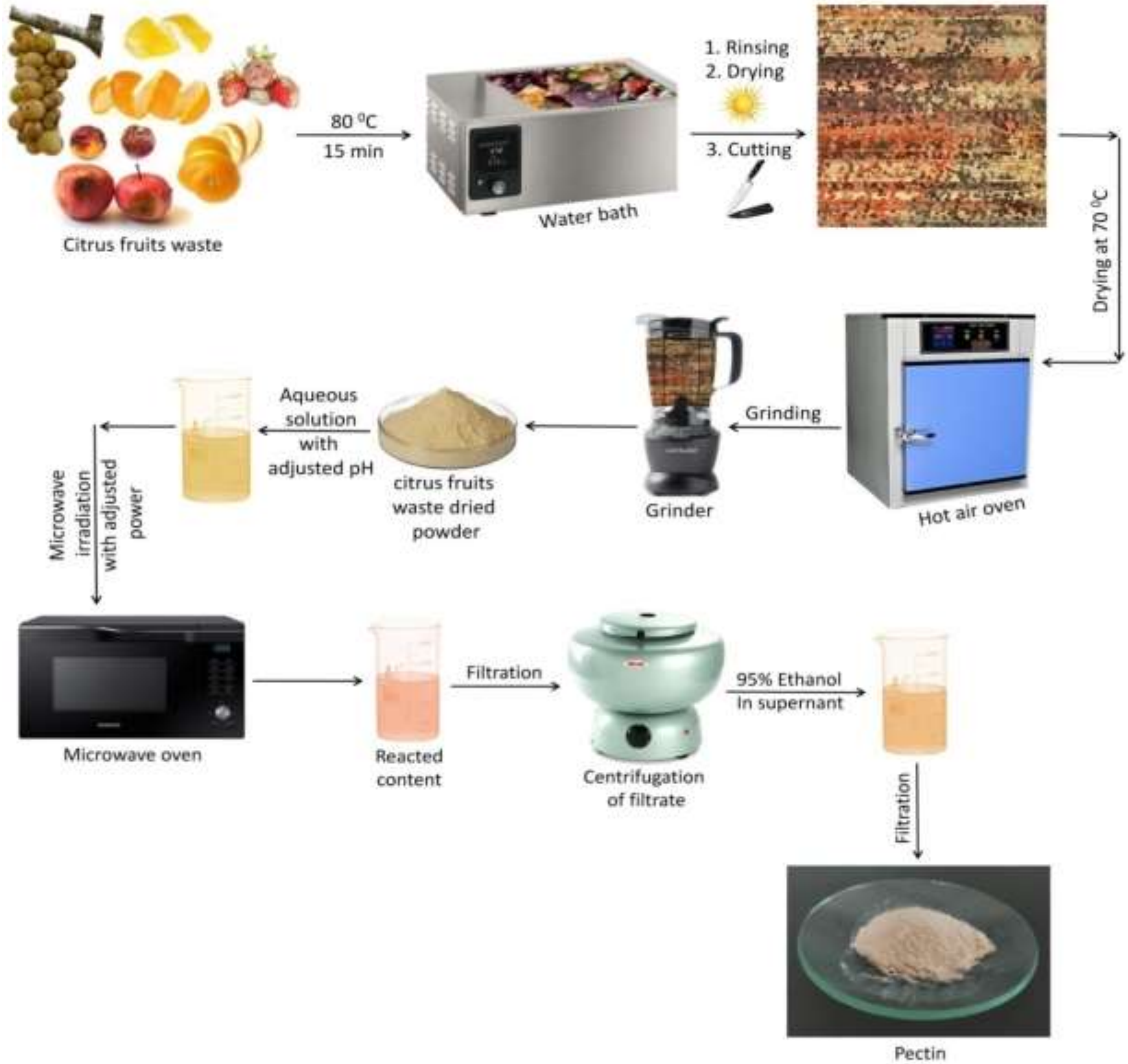

Fig.1. Experimental design for pectin extraction from citrus fruits waste

2.3. Determination of pectin yield

The extracted moist pectin after being dried in hot air oven to attain constant weight, the pectin yield (% w/w) was calculated using the formula given below [39].

$Y (\%, w/w) = (m_0/m) \times 100$

Where, m_0 (g) is the weight of dried pectin

m (g) is the weight of citrus fruits waste powder

2.4. Characterizations

SHIMADZU FTIR-8400S was employed for the FTIR spectral analysis of extracted pectin between 400 cm^{-1} and 4000cm^{-1}. The calibration of CHNSO analyzer (Horriba EA3000) with standard sulphanilamide prior to the extracted pectin sample elemental analysis was done.

III. DISCUSSION & RESULT

3.1. Optimization of microwave assisted extraction conditions

The efficiency of pectin extraction is affected by factors such as pH of solution, S/L ratio (solid-liquid ratio), microwave illumination power and illumination time. Compared to methods of previous study, (1.5 pH of reaction solution adjusted with 0.5 M HCl, keeping solid–liquid ratio 1:50 [39] and 125s illumination time at decreased pH values the improved extraction efficiency of pectin by raising microwave power from 160–480 W [40]) the experimental results of present study were presented as follows. The experimental results of pectin yield extracted from citrus fruits waste using microwave with solid-liquid ratio 1:30 was shown in Table-1 demonstrated that, Low power (180W), Low Time (60 sec.) and Low pH (1.0) gives Minimum %Yield (16.35%); High Power (450W), Optimum Time (120 sec.) and Low pH (1.0) gives Optimum %Yield (25.45%); Optimum power (300W), Optimum Time (120 sec.) and Optimum pH condition (1.5) improved extraction efficiency giving Maximum %Yield (34.55%).This accelerated pectin extraction by applying optimum power (300W) is related to the direct effect of this condition on plant material more effectively rather than 180W and 450W which loosens the cell wall matrix and thus the skin tissues are extensively and rapidly opened up and help to increase the interaction between source material and extraction media improving the extraction efficiency. However, the optimum microwave exposure time i.e. 120 sec. retard the pectin degradation. The optimum strength of acidic solvent permits coherence with the insoluble pectin to hydrolyze insoluble pectin into soluble pectin and increases pectin yield significantly and reached to maximum by employing pH condition 1.5. However, fluctuation of pH valuefrom 1.5, retarded the pectin release might be due to the accumulation of pectin. In addition pectin yield can be improved for the higher concentration gradient of dissolved pectin in S/L ratio 1:30 than that of S/L ratio 1:50 [39].

Table-1 Optimization of pectin yield

Sr. No.	Volume (ml) Containing 1 (gm) citrus fruit waste powder	pH	Time (sec.)	Power (W)	Weight of dried pectin (gm) m_0	%Yield
1		**1.0**			**0.1635**	**16.35**
2		1.5	**60**		0.2895	28.95
3		2.0			0.1845	18.45
4		1.0			0.2475	24.75
5		1.5	120	**180**	0.3315	33.15
6		2.0			0.2685	26.85
7		1.0			0.2055	20.55
8		1.5	180		0.3105	31.05
9		2.0			0.2265	22.65
10		1.0			0.1775	17.75
11		1.5	60		0.3035	30.35
12		2.0			0.1985	19.85
13		1.0			0.2615	26.15
14	30	**1.5**	**120**	**300**	**0.3455**	**34.55**
15		2.0			0.2825	28.25
16		1.0			0.2195	21.95
17		1.5	180		0.3245	32.45
18		2.0			0.2405	24.05
19		1.0			0.1705	17.05
20		1.5	60		0.2965	29.65

21		2.0		450	0.1915	19.15
22		**1.0**	**120**		**0.2545**	**25.45**
23		1.5			0.3385	33.85
24		2.0			0.2755	27.55
25		1.0	180		0.2125	21.25
26		1.5			0.3175	31.75
27		2.0			0.2335	23.35

3.2. Characterization

3.2.1. FT-IR characterization of Pectin

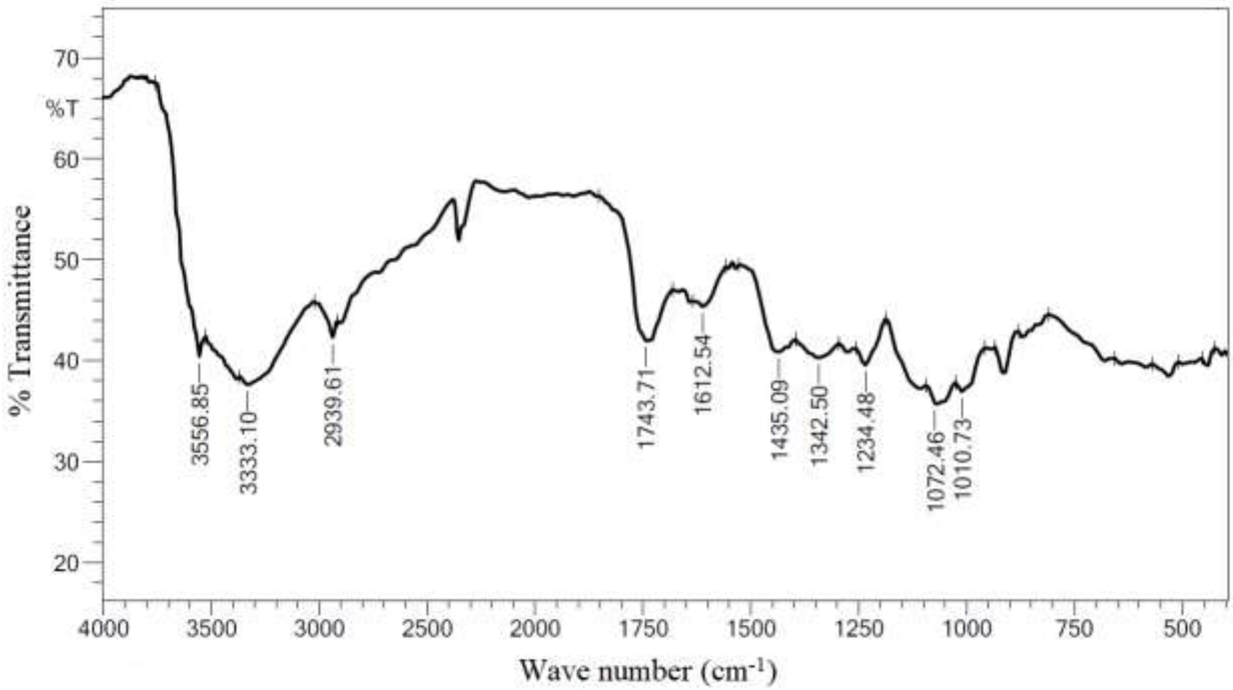

Fig.2. FT-IR spectra of extracted pectin from citrus fruits solid waste

As shown in Fig. 2 the FTIR spectra of extracted pectin reveals that 3557 and 3333 cm^{-1} bands characterize O-H stretching absorption including intramolecular hydrogen bonding. The 2940 cm^{-1} band arises due to C-H stretching absorption including -CH- and $-CH_3$ stretching vibrations. The 1744 cm^{-1} band characterize C=O stretching vibration of COOH group and methyl ester group, whereas band at 1613 cm^{-1} characterize stretching vibration of carboxylate (-COO-) group. The bands at 1435 cm^{-1}, 1343 cm^{-1} and 1234 cm^{-1} arise from bending of C-H in pyranoid ring, O-H bending (in a plane) and $-COCH_3$ stretching respectively. Glycosidic bond (C-O) stretching and C-C bond stretching of pyranoid ring are characterized by bands 1072 and 1011 cm^{-1} respectively in the finger print region [41]. These reflect composition of pectin.

3.2.2 Table-2 Elemental Analysis of Pectin

Sr. no	Type	Name	C %	H %	N %	S %	O %	Weight (mg)
1.	Standard	Sulphanilamide	42.05	4.74	18.75	21.52	12.94	0.985
2.	Standard	Sulphanilamide	40.91	4.67	15.81	18.58	20.03	1.125
3.	Sample	Pectin	41.02	4.69	0.01	0.02	54.26	1.105

Elemental analysis calculated value for $(C_{13}H_{18}O_{13})_n$ (Pectin): C (40.84%), H (4.71%), O (54.45%) are matched very well with the experimental elemental analysis value: C (41.02%), H (4.69%), O (54.26%) are shown in Table-2.

IV. CONCLUSION

Three factors such as pH, illumination time (sec.), and microwave power (W) were successfully employed for the optimization of pectin yield. The extraction efficiency of pectin from citrus fruits solid waste was influenced significantly by optimizing these process variables of an efficient, time saving and eco-friendly microwave technique. The optimal conditions were as follows: 300W microwave power, solid-liquid ratio 1:30, 120s illumination time and pH of 1.5 with maximum pectin yield (34.55%).

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