

RESEARCH ARTICLE

PREPARATION AND CHARACTERIZATION OF PECTIN FROM PEEL OF KABAD (*CITRUS MEDICA*) FRUIT IN SULAIMANI CITY, IRAQI KURDISTAN REGION

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Abstract

This research study aimed at comparing the characteristics of pectin that is prepared from peel of Kabad (*Citrus medica*), Kabad belongs to the citrus family and has a thicker skin that can be used as a source of pectin. It was extracted using different acids (HCl, HNO₃, and citric acid) at different temperatures (50°C, 70°C, and 100°C) using sun-dried and dried (at 130°C) peels (pH=2.0) and precipitated by ethanol. The color of the pectin was brown. The pectin yield in sun-dried peel is from (10%-40%) while dried at 130°C was (28%-65%) and the best extraction condition was using citric acid at 100°C using dried peel at 130°C. The moisture content is 13%, methoxyl content is 1.9%, degree of esterification is 11.75%, and equivalent weight was 725 g/mol by HCl as the extracting solvent.

Keywords: Citrus medica peels, pectins, methoxyl, sundried

Introduction

Citrus medica, commonly known as a citron in English, but in Kurdistan city Iraq known as Kabad (figure-1), is a small tree having large fruit (20-22.5 cm long) resembling a pineapple in shape - citrus fruits which consist of two parts, namely the peels (rind/skin) which is the important part of the citron and the pulp. These two parts are easily separated from each other with the pulp serving as the edible part of the fruit while the peels are a good source of pectin (2). Pectin (3) is a structural heteropolysaccharide contained in the primary cell walls of terrestrial plants. It was first isolated and described in 1825 by Henri (4,5). It is produced commercially as a white to light brown powder, mainly extracted from citrus fruit and is used in food as a gelling agent, particularly in jams and jellies. It is also used in fillings, medicines, sweets, as a stabilizer in fruit juices and milk drinks,

and as a source of dietary fiber. Pectin is a polysaccharide widely used in food and pharmaceutical industries. It is used as thickening and gelling agents (6). Its medical uses are antidiarrhea, detoxification, and blood glucose lowering (7).



Fig-1-Kabad (*Citrus medica*) fruit in Sulaimani city

According to Levigne et al(8), pectins are methylated ester of polygalacturonic acid that contains 1,4-linked -D-galacturonic acid residues. Yeoh et al(9) had reported that these polysaccharides consist of 300-1000 chains of galacturonic acid units. High methoxyl (HM) pectins have more than half the carboxyl groups as methyl esters and form gels in the presence of high sugar concentrations and acid (e.g. jams, jellies, marmalades). Low methoxyl (LM) pectins have less than half the carboxyl groups as methyl esters⁹.

Materials and Methods

Sample collection and drying

kabad(citrus medica) fruit harvested from sulaimani city, Iraq during 2014. The fruit was cut into four parts and the peel removed (a soft white substance inside the skin of citrus medica fruits), then the peel was further cut into smaller pieces for easy drying and washed with large quantity of water to remove the Glycosides the bitter taste of the peel and then weighed and air dried.

Extraction of pectin

The 5g dried peel transferred into a beaker containing 250 mL of water, the PH was adjusted to 2.0 with hydrochloric acid, nitric acid and citric acid respectively the fruit was then heated to 50, 70, 100°C for 1h, peel was removed from the extracts by filtering, filter allowed to cool at 4°C temperature(11). The extracted pectin was precipitated by adding 95% ethanol stand for 30 min to allow the pectin float on the surface. The extracted pectin was purified by washing with ethanol. The resulting pectin was weighed and was air dried. The yield of pectin was calculated(12)

$$y_{pec}(\%) = Y = 100 \times \left[\frac{P}{B_i} \right]$$

y_{pec} is the extracted pectin yield in percent (%),
P is the amount of extracted pectin in g and B_i is the initial amount of ground peel (5g).

Equivalent weight determination

Equivalent weight it is determined by titration with NaOH to PH 7.5 using phenol indicator(13). 0.5g of pectin was weighed then diluted with 5ml of ethanol 1g of NaCl was added also 100ml water and phenol red indicator then titrate with 0.1N NaOH until the

color was change (13)

$$\text{Equivalent weight} = \frac{\text{weight of sample} \times 1000 / \text{ml of alkali} \times \text{Normality of alkali}}$$

Moisture content determination

A dried empty Petri dish was dried in an oven, cooled in a desiccators and weighed 1 gm of the pectin sample was transferred into the crucibles in the oven for 1 h the Petri dish was removed, cooled in a desiccators and weighed content (14). This solution used for methoxyl content.

$$\text{Moisture Content (\%)} = \left(\frac{\text{Weight of the Residue}}{\text{Weight of the Sample}} \right) \times 100\%$$

Methoxyl Content

The methoxyl contents were determined by adding 25 ml of 0.25 N NaOH to the neutral solution then stand for 30 minutes at room temperature. 25 ml of 0.25N HCl was added and titrated with 0.1N NaOH (15).

Degree of esterification

The degree of esterification (DE) of pectin was determined according to the formula given below (16)

$$\%DE = \frac{176 \times \text{CH}_3\text{O}\% \times 100}{31 \times \text{AUA}\%}$$

$$\%AUA = \frac{\text{meq of NaOH} \times 31 \times 100}{(176 \times 100) / Z}$$

176 = Mwt of AUA (Anhydrouronic acid)
Z = wt of sample / mequivalent weight of alkali for free acid + meq of alkali of methoxyl

Results and Discussion

This study investigated the effect of temperature and acid condition on the extraction of pectin from fruit peel of citrus medica, the color of it is important the pectin was dried by sun has light brown but the pectin dried at 130°C was deep brown that is affects of the gel produced. Table-1- shows amount of moisture content in pectin in different temperature and acid from 9.0%-17.0% using sun dried comparing with dried at 130°C ranges from 10.0%-18.0% which is higher.

The percentage of yield show more when dried at 130°C if compared with sun dried peel. The highest yield was 65% that obtained from dried at 130°C

using citric acid and heated at 100°C but for sun dried peel the highest yield 40% using citric acid at 100°C Table-2-,figure-1- citric acid was best for extraction of pectin.This agreement with results reported by Virk and Sogi (9) has been shown that acid type strongly influences the macromolecular and gelling properties of isolated pectins, with citric acid being the least pectin degrading (depolymerising and deesterifying) extracting agent it mean the pectin isolates with the best gelling properties .

The value of equivalent weight was highest (725g/mole) table-3-using citric acid and dried at

130°C but for sun dried the value was(650g/mole) using nitric acid and sun dried.The methoxyl content is an important for ability of pectin to form gels. Table-4-,Figure -2-the methoxyl content when dried peel at 130°C was highest (3.8%) by HCl at 50°C but for sun dried the highest value was (1.60%) by HNO₃ at 100°C, the methoxyl content in pectin between (1.1-1.6%)for sun dried and (1.2-3.8%) for dried at 130°C this mean low methoxyl pectin therefore the degree of esterification was low than 50% table-5- indicating that the pectin was good in term of quality .

Table-1- Moisture content of pectin in different temperature and acid extracting

Acid	T ⁰ C	Sun dried	Dried at 130 ⁰ C
HCl	50	13	14
	70	17	18
	100	16	15
HNO ₃	50	13	15
	70	12	17
	100	11	18
Citric acid	50	9	10
	70	12.5	12
	100	13	13

Table -2- %pectin in different temperature and acid extracting

Acid	T ⁰ C	%pectin (Sun dried)	%pectin (Dried at130 ⁰ C)
HCl	50	18	28
	70	20	30
	100	10	38
HNO ₃	50	20	48
	70	30	45
	100	35	50
Citric acid	50	25	55
	70	35	58
	100	40	65

Table -3- Equivalent weight in different temperature and acid extracting

Acid	T ⁰ C	Equivalent weight (Sun dried)	Equivalent weight (Dried at 130 ⁰ C)
HCl	50	280	550
	70	270	555
	100	260	725
HNO ₃	50	610	500
	70	615	600
	100	650	550
Citric acid	50	200	220
	70	210	215
	100	200	225

Table-4- methoxyl content of peel drying in different temperature and acid extracting

Acid	T °C	% methoxy content (Sun dried)	% methoxyl content (Dried at 130 ⁰ C)
HCl	50	1.2	3.8
	70	1.25	3.5
	100	1.4	2.9
HNO ₃	50	1.45	1.2
	70	1.5	1.3
	100	1.6	1.5
Citric acid	50	1.1	1.7
	70	1.3	1.8
	100	1.5	1.9

Table-5- Degree of esterfication(DE) in different temperature and acid extracting

Acid	T ⁰ C	(DE) (Sun dried)	(DE) (Dried at 130 ⁰ C)
HCl	50	12	38
	70	13	32
	100	14	30
HNO ₃	50	14	12
	70	15	15
	100	16	16
Citric acid	50	10	17
	70	12	17.2
	100	15	11.75

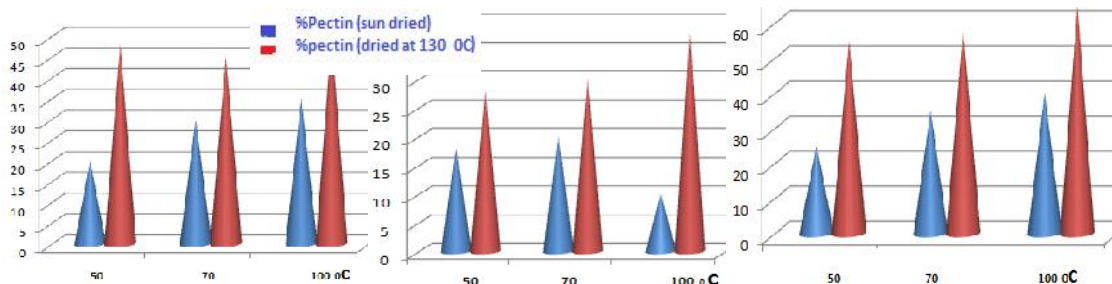


Fig-2- %pectin in different temperature and acid extracting (HCl,HNO3and citric acid)respectively

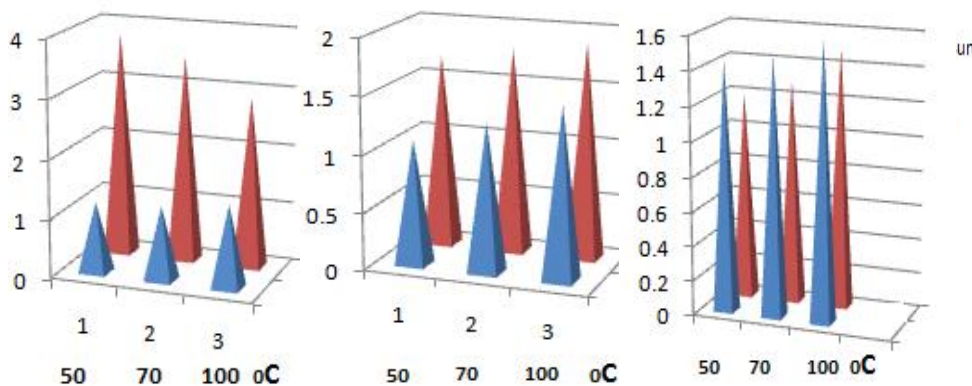


Fig -3- methoxyl content of peel drying in different temperature and acid extractin (HCl,HNO3 and citric acid)respectively

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