

Extraction of Pectin from Eggplant Peels and Calyxes and Studying its Physicochemical and Functional Properties

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Abstract

The current study aimed to extract pectin from eggplant peels and calyxes and studying chemical and physical composition for its, determine the best solution for extraction, which included (citric acid, hydrochloric acid, oxalic acid) at a temperature of 90°C and an extraction time of 90 minutes, as well as studying the physicochemical and functional properties of the extracted pectin. The results of the studying indicated that the highest percentage of pectin extraction yield was obtained when using citric acid with a concentration of (27.3) %, followed by hydrochloric acid (26.1%), while oxalic acid recorded the lowest extraction yield (23.1%). Based on the results of the best extraction yield, the pectin extracted with citric acid solution was used to study its chemical properties, as the degree of esterification was (61.33) %, the methoxyl content (5.76) %, the reductive force (1.38) eq/g, galacturonic acid (64.9) %, and the equivalent weight (1898.1) g/eq. The physical properties of pectin extracted from eggplant peels and calyxes were also estimated, including relative viscosity (1.1343), molecular weight (229.79) kDa. As for the functional properties of pectin extracted from eggplant peels and calyxes, which included water holding capacity (3.3) g/g pectin, fat holding capacity (2.86) g/g pectin, the foam capacity rate for the three concentrations was (71.3)%, while the foam stability rate was (31.5) % , while the rate of emulsification stability for the three concentrations was (75.8) %.

Keywords: *Pectin, Eggplant peels and calyxes, Pectin Extraction Yield, Functional Properties of Pectin.*

Introduction

Pectin is a widespread food ingredient with high nutritional value that is used as a gelling agent and is found in all plant cell walls that are widely found in nature, pectin is also found in plants united with polysaccharides such as cellulose and hemicellulose, and pectin is found in plants in the form of calcium pectin with a high molecular weight, Pectin was invented by the French scientist Henri Procono in year 1825 for the first time, as it has functional importance in plants such as its ability to retain water and ionic transport (14).

The pectin is one of the economically important materials in the food industry, as it is included in the manufacture of jams, fruit juices, jellies, and sweets, and it is also included in some pastries. There is also a major use of pectin in the dairy industry, especially low-fat to give it a cohesive texture, it is also used in the manufacture of medicines and cosmetics as a material for treating wounds and physical deformities and many other industries (4).

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The peel and calyces of eggplant are among the main by-products of eggplant, as these wastes contain materials of high nutritional value, as the eggplant peel contains a lot of nutrients and antioxidant phenolic compounds. Moreover, the peel and calyces are sources of plant fibers such as pectin and cellulose, so these by-products are inexpensive and available sources, as these products contain high amounts of moisture, reaching about (90%) (35). In addition to the organic components, which means that they are subject to rotting easily and therefore, if they are disposed of incorrectly, they will pose serious threats to the environment, so some studies have been conducted to exploit these residues in the food industry to benefit from them by extracting pectin materials from them and introducing them into other food products because of its many benefits, the pectin is included as a thickener in jams, baked goods and pastries, and is included in milk products such as yogurt (22).

The process extracting of pectin is a multi-stage process that includes chemical and physical analyses to extract pectin from plant waste (33). The extraction process is influenced by various factors including temperature, pH, and time, and then the pectin that isolated, purified, and dried (24).

pectin is extracted in several ways, including the traditional method or by modern methods, including Ultrasound, Microwaves, or Enzymatic extraction (21). The pectin is extracted in the traditional way by using dilute acids such as hydrochloric acid, nitric acid, sulfuric acid, phosphoric acid, citric acid or chelating agents such as potassium oxalate or using dilute sodium hydroxide or cold, hot extraction (3).

Materials and methods

1- Methods of Extraction

A- Extraction by Citric Acid

Method (19) was followed by adding (5)g of powdered eggplant peels and calyces to 250 ml of (0.2)M citric acid at pH (1.5), then the mixture was heated to 90 °C for 90 minutes on the heater and magnetic stirrer, the mixture was filtered using a wet cloth and the pectin was precipitated by mixing the filtrate with a similar volume of ethanol 99% and left in the refrigerator for 60 minutes, then filtered and dried the precipitate at 50 C ° for 24 hours and kept in the freezer at a temperature of (-18)°C until use.

B- Extraction by Hydrochloric Acid

The method (19) was followed, with the same steps followed by citric acid extraction, except for the replacement of citric acid with hydrochloric acid at a concentration of (0.5)M.

C- Extraction by Oxalic Acid

Method (28) was followed by adding (5)g of peels and dry calyces in 100 ml of 2% oxalic acid and heating the mixture to 90°C for 90 minutes using a heater and magnetic stirrer, then the same extraction steps were followed by citric acid.

2- Chemical Properties

A- Esterification Degree

The degree of esterification was estimated according to method (17) by moistening (0.5) g of dried pectin with (5) ml of 99% ethanol in a conical flask and dissolving it in (20)ml of distilled water at a temperature of 40°C with stirring using a magnetic stirrer and titrating the solution using (0.1)M sodium hydroxide until a pale pink color appeared and the final volume was recorded. The degree of esterification was calculated by applying the following equation:

$$\text{Esterification Degree(ED) \%} = \frac{\text{Final volume of NaOH}}{\text{Primary volume} + \text{Final volume}} \times 100$$

B- Methoxyl Content

Content of Methoxyl was estimated according to method (15) by wetting (0.5)g of pectin in 5 ml of ethanol (99%), then 1g of sodium chloride was added, then 25 ml of (0.1)N hydrochloric acid was added, then it was titrated with (0.1)N sodium hydroxide with continuous stirring until reaching a pH of 7.5 and the final volume was recorded. Calculate the methoxyl content of pectin by the following equation:

$$\text{Methoxyl Degree} = \text{Final base volume} \times \text{Base Molarity} \times 31 \times 100 / \text{sample weight (g)}$$

C - Reductive Force

The reductive force of the pectin solution was estimated according to the method (29) by titration with ferric cyanide and the number of ferric cyanide was calculated from the equation:

$$\text{Potassium cyanide number (Eq/g)} = \frac{(A-B) \times \text{Sodium Thiosulfate Molarity} \times 10}{\text{Dry sample weight (g)}}$$

whereas:

A: Represents the number of milliliters of sodium thiosulfate consumed in the control sample.

B: Represents the number of milliliters of sodium thiosulfate consumed in the samples.

D - galacturonic acid

Followed the method described by (12), 0.5 ml of pectin solutions were placed in test tubes, 6 ml of concentrated sulfuric acid was added to it at a ratio of (6:1) acid: distilled water, then 0.2 ml of 0.1% carbazole solution, the tubes were shaken hard after being tightly closed and put in a water bath at 75 °C for 20 minutes, then they were quickly cooled and the resulting color intensity was estimated at a wavelength of 525 nm.

3- physical properties

A – Viscosity

Viscosity was estimated according by method (38), dissolving 1 g of dry pectin in 100 ml of distilled water, then the viscosity was measured using a capillary tube at a temperature of 25°C and a volume of 15 ml of the prepared pectin solutions, then according to the time required for the flow of the pectin solution, and distilled water was used for comparison, and the viscosity of the prepared pectin solutions was extracted according to the following equation:

$$N_r = d_1 t_1 / d_2 t_2$$

B- Molecular weight

According the method (31) molecular weight was estimated by dissolving 0.1 g of pectin in 100 ml of sodium hexameta phosphate solution prepared at a concentration of 1%, and then filtering the solution with filter paper, relative viscosity of the prepared pectin solutions and the sodium hexameta phosphate solution was measured with a capillary tube, after which the specific viscosity (NSP) was extracted from the equation:

$$\text{Molecular Weight} = \text{Real Viscosity} / C$$

C: galacturonic acid concentration

4- Functional features

A- Water Holding Capacity

Water holding capacity of pectin was estimated by the method mentioned (6) and the water holding capacity was calculated through the following equation:

$$\text{Water holding capacity (g/g of pectin)} = \frac{\text{Wet sample weight} - \text{dry sample weight}}{\text{dry sample weight}}$$

B- Fat Holding Capacity

Method (6) was used to determine the ability of pectin powder to hold fat, as this property was estimated with the same steps followed in estimating the ability to bind water, but by replacing 10 ml of distilled water with sunflower oil, and the ability to bind fat was expressed by the number of grams of oil absorbed per gram of the sample.

$$(\text{g of fat/g dry weight}) = \frac{\text{Weight of the tube with the fat} - \text{absorbing sample} - (\text{Weight of the tube with dry sample})}{\text{Weight of dry sample}}$$

C - Foam and Stability

Method described by (32) was followed in estimating the ability of pectin to form foam and its stability, as different concentrations of pectin were prepared and included (0.1, 0.3, 0.5) g with 100 ml of distilled water, and then the mixture was mixed well by a Vortex device at a speed of 1500 rpm and the foam was measured after a time period of 10 seconds, while the stability of the foam was estimated after 30 minutes. The foam capacity and stability were calculated according to the following equations:

$$\text{Foam Stability \%} = \frac{\text{Volume of Foam after 30 minutes}}{\text{Total Volume of suspension}} \times 100$$

$$\text{Foam \%} = \frac{\text{the first volume of foam}}{\text{Total volume of the suspension}} \times 100$$

D- Emulsification and Stability

The method described by (32) was followed in estimating the ability of pectin to form emulsifiers by preparing solutions with concentrations of (0.1, 0.25, 0.5) % (w/v) in distilled water, and the pH of the extracted pectin solutions was adjusted at pH=7, and then the emulsions of pectin/sunflower oil were mixed in ratios (160/40) (v/v) for each of the different concentrations of pectin by the vortex device at a speed of 8000 rpm for 10 minutes. The emulsification was calculated as a percentage of the volume of the emulsion to the total volume. The stability of the pectin was also estimated by the same method, but at a temperature of 85 °C for 30 minutes in a water bath, after which it was cooled. The percentage of the emulsion and its stability were calculated using the following equations:

$$\text{Emulsion Capacity (\%)} = \frac{\text{Volume of Emulsion}}{\text{Total Volume}} \times 100$$

$$\text{Emulsion Stability (\%)} = \frac{\text{Final Volume of Emulsion}}{\text{Volume of First Emulsion}}$$

Results and Discussion

1- Determining the efficiency of the solutions used to extract pectin from Eggplant peels and calyxes

The results showed in Figure(1) the yield percentages of pectin extracted from eggplant peels and calyxes, as it was noted that the extraction with citric acid solution gave the highest yield percentage compared to the rest of the solutions used, reaching (27.3)%. (1) pointed out that when extracting pectin from lemon peels by citric acid, the percentage of pectin yield was (28.4)%. The results also show that citric acid gave the highest yield compared to other acids, and the reason is that citric acid is considered a natural acid found in many plants, and the temperature and time of extraction and the optimum pH concentration is (1.5) and these conditions make it suitable for extraction (26).

As for the extraction with hydrochloric acid, the percentage of yield of pectin was (26.1) %, shown by (1) that when extracting pectin from citrus peels using HCL acid, the percentage of pectin was (25.1)%, and it also indicated that the percentage of pectin extracted with the same acid was (15.2)% from pomegranate peels. The reason for the discrepancy in the yield ratios may be due to the fact that acid extraction leads to the decomposition of the proto pectin present in the sample and its transformation into soluble pectin, as well as the extraction of free pectin substances and their transformation into small pectin particles resulting from partial decomposition, and thus an increase in their solubility to a degree so that they do not precipitate when alcohol is added (19).

When extracting pectin by oxalic acid, the percentage yield of pectin from eggplant peels and calyxes was (23.1) %, (1) showed that when extracting pectin from orange peels by means of Oxalic acid, the extraction rate was (23.2%).

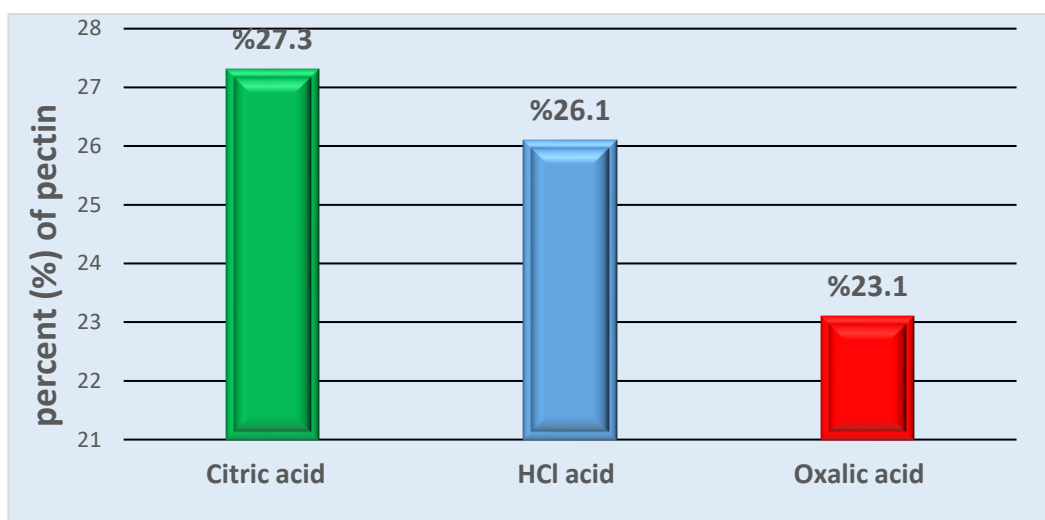


Figure 1. The percentages of pectin yield using different extraction solutions

2- Chemical Properties

A- Esterification Degree

Table(1) showed that the percentage of pectin extracted from eggplant peels and calyxes by citric acid method was (61.33)%, (1) indicated that the pectin extracted from pomegranate waste was (66)%, while (20) indicated that the percentage of esterification in apple pomace was (60.49)% when it was used at a temperature of 95°C and at a time of one hour, while it increased to (63.73) %, when using the autoclave temperature in the extraction process. Studies indicated that the increase in the percentage of the degree of esterification in pectin (60)% is one of the desirable chemical properties of pectin in the food industrial fields because it is related to the quality of the resulting gel and the speed of its formation (8).

B- Methoxyl Content

The results in Table (1) showed that the percentage of Methoxyl groups of pectin extracted from eggplant peels and calyxes, where the percentage of Methoxyl content was (5.76) %. (20) showed that the Methoxyl content of two types of apple fruit pomace (*Spondias dulcis*, *Malus pumila*) is (6.21, 4.82)%, respectively. The number of Methoxyl groups and their distribution on galacturonic acid deposits is one of the important and main factors that determine the chemical, physical and functional properties of pectin materials such as their solubility in water, gelation property of pectin also depends very much on the degree of methylation (DM) degree of methylation, on the basis of which the pectin is divided into two main parts: High Methoxyl Pectin (50-80%) (HMP) and it depends on pH

is less than 3.6 and sucrose is more than 50% for gelation, the Low Methoxyl Pectin (25-50%) (LMP) depends on the formation of the gel on the addition of calcium ions (37).

C - Reductive Force

The results showed in Table (1) that the reductive force of pectin extracted from eggplant peels and calyxes is in the form of ferric cyanide number, which indicates that the amount of aldehyde groups at the ends of the chains of pectin substances, as the value of the reductive force was (1.38) equivalent / g of pectin extracted with citric acid. (1) found that the pectin extracted from citrus residues had values for the reductive force amounted to (1.36- 2.55) Eq/g by different temperatures.

D - Galacturonic Acid

Table (1) showed that the percentage of galacturonic acid in the pectin extracted from the peels and calyxes of eggplant extracted by the citric acid method amounted to (64.9) %. (10) concluded that the percentage of galacturonic acid ranged between (42 - 69.1)% in the pectin extracted from banana waste. (36) mentioned that the percentage of galacturonic acid reached (59.9)% in the extracted pectin of apple pomace extracted by the acid method, and indicates that the content of galacturonic acid is to the degree of purity of pectin, and that the percentage of this acid is less than (65)% indicates the possibility of the presence of precipitated sugars and proteins with the pectin (16).

Table 1: Chemical properties of pectin extracted from eggplant peels and cones

Chemical Properties	(%)
Esterification Degree	61.33
Methoxyl Content	5.76
reductive force Eq/g	1.38
Galacturonic Acid Content	64.9

3- Physical Properties

A - Relative viscosity

The results showed in Table (2) the relative viscosity of pectin extracted from eggplant peels and calyxes by acid extraction method amounted to (1.1343), (1) indicated that the value of relative viscosity in citrus peel pectin reached (1.18) under extraction conditions of 90°C and an extraction time of 90 minutes, while (27) found that the value of relative viscosity of pectin extracted from watermelon peels amounted to (1.21). As there are many factors that affect the viscosity of pectin materials, such as molecular weight, degree of esterification, pH, and the presence of calcium ions (9).

B- Molecular weight

Table (2) showed the results of the value of the molecular weight of pectin extracted using citric acid from eggplant peels and calyxes amounted to (229.79) kilo Dalton. It was mentioned by (7) that the temperature, pH, and the duration of extraction are among the important factors that have an impact on the value of the molecular weight of pectin, and the time factor is one of the most important factors that can affect this result, as the value of the molecular weight of pectin decreases whenever the extraction time exceeds 60 minutes, due to the decomposition of the pectin materials. (39) indicates that the value of the molecular weight of pectin depends on the content of viscosity and galacturonic acid, as the higher the viscosity, the greater the value of the molecular weight of pectin. Another study found that (11) estimated the molecular weight of passion fruit pectin between (130-181) kilo Dalton.under acid extraction conditions and at pH (1). (36) obtained in his study

when estimating the molecular weight of apple pomace pectin extracted acidically and at pH(2) and at a temperature of 85 °C with a time of 180 minutes, that the value of the molecular weight of the pectin was 331 kilo Dalton.

Table 2: Physical properties of pectin extracted from eggplant peels and calyxes

Physical Features	Pectin Extracted with Citric Acid
Relative Viscosity	1.1343
Molecular Weight /kDa	229.79

4- Functional Characteristics

A- Water Holding Capacity

Table (3) showed that the ability to hold water for pectin extracted from eggplant peels and calyxes amounted to (3.3) g water /g pectin. (25) found that pectin extracted from tomato peels has a water-holding capacity of (3.5) g water / g pectin. (2) studied the functional properties of pectin from tomato waste, as the water-holding capacity reached (3.2)g water /g pectin powder. The higher quality of pectin and also the ability to bind water is of great importance in improving the sensory characteristics represented by the texture and appearance of the frozen food by reducing the rate of evaporation and the formation of small ice crystals (34).

B- Fat Holding Capacity

The results in Table (3) showed that the fat holding capacity of pectin extracted from eggplant peels and calyxes amounted to(2.86) g fat/ g pectin. (30) studied the functional properties of pectin extracted from (Averrhoa bilimbi) fruit plant, as the fat holding capacity amounted to (2.7) g fat/g pectin, while (13) mentioned that the pectin extracted from the bean plant (*Parkia speciosa*) has a fat holding capacity amounting to (3.9) g fat/g pectin, the reason for the difference in the fat-holding ability of the extracted pectin types is attributed to the protein percentages and the presence of amino acids that contain the active aggregates, as this estimate is important for identifying the ability of the pectin molecule to absorb oil, because this molecule is colloidal, so the greater the percentage of pectin ability to hold fat, the higher the quality of the pectin, meaning that the benefit of the phenomenon of pectin bonding with fat is to retain the distinctive flavors when food is exposed to high cooking temperatures, and the ability to bind to fat increases the retention of flavors because most of them are soluble in fat and improve the taste qualities of the product and increase the feeling of a greasy taste in the mouth (5).

C- Emulsification and Stability

Table (3) showed the results of the emulsification property of pectin extracted from eggplant peels and calyxes, where the emulsification rate for the three concentrations was (75.8) %. The emulsifying stability of pectin extracted from eggplant peels and calyxes reached for the three concentrations (0.1, 0.25, 0.5) % (72.4, 75.9, 79.2) %, respectively, Figure (2), the stability of the emulsification increased with increasing the concentration of the extracted pectin, and the pectin extracted from eggplant peels and calyxes was stable when heated at 85 °C for 30 minutes for the same concentrations. (18) performed that the pectin of eggplant peels and calyxes has an excellent emulsification stabilizing activity, and the reason is attributed to the amount of protein present in the pectin through which it leads to an increase in surface activity.

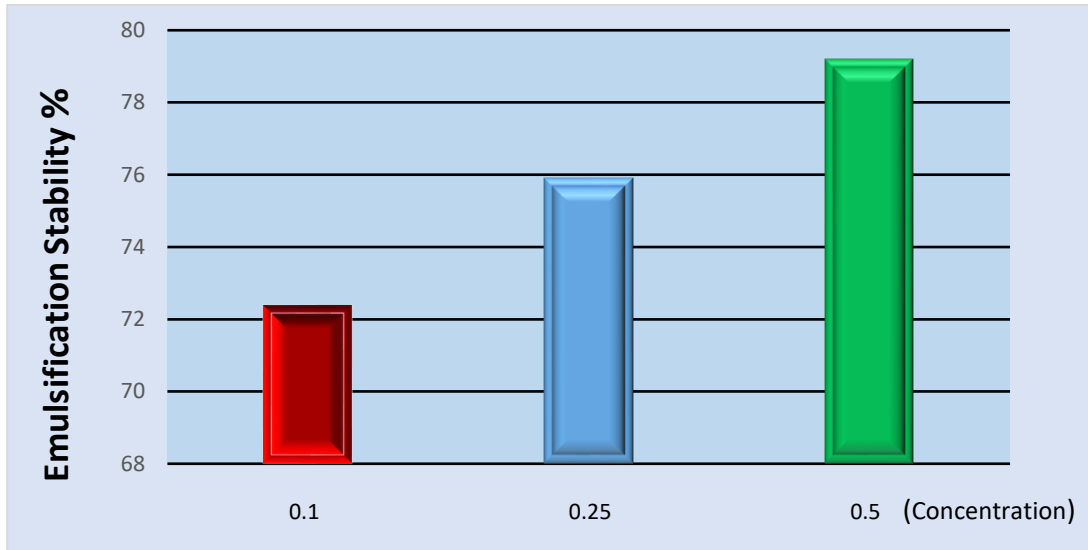


Figure (2). The relationship between pectin concentration of eggplant peels and calyces and emulsification stability

D - Foam and Stability

Figure(3) showed the relationship between the different concentrations of pectin solution extracted from eggplant peels and calyces, which showed a significant decrease in the foaming capacity when increasing the concentration (0.1, 0.3, 0.5)% and at room temperature, reaching (68,71.3,74.6)% respectively. The average foaming capacity for the three concentrations was (71.3) % (Table 3). Indicated percentage the foam capacity of pectin peels and calyces eggplant that the concentration of pectin has an opposite effect on the foam, as the results, the study showed that increasing the concentration decreased the entry of air during whipping process, which led to a decrease in foam capacity. Therefore, the higher pectin concentration, the lower foam capacity, as the foam capacity has a great effect on general volume of many food products, it gives an excellent feeling and sensation in the mouth and others (23).

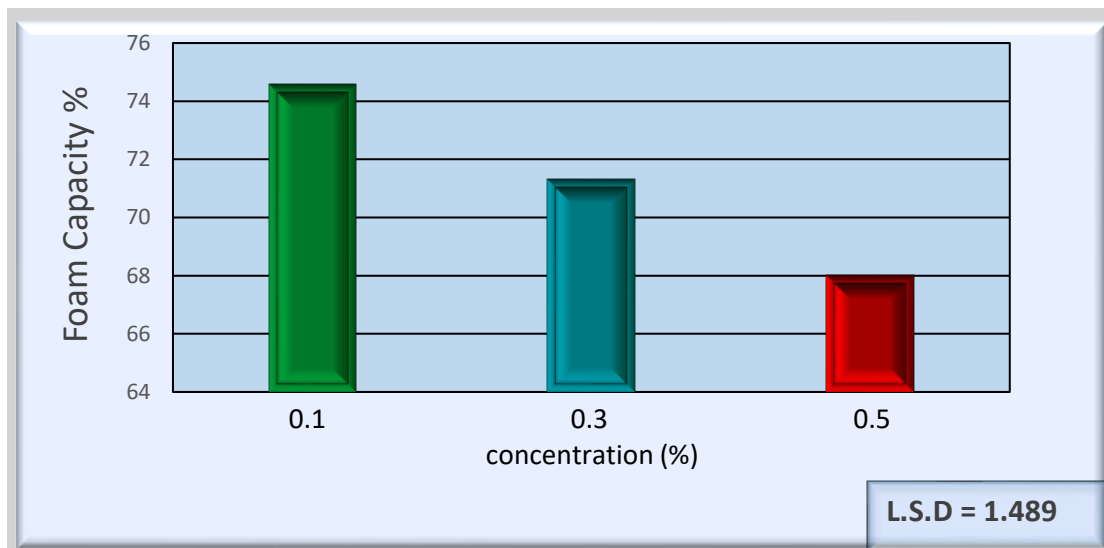


Figure (3). The relationship between pectin concentrations extracted from eggplant peels and calyces and foam capacity.

The foam stability of the different concentrations (0.1, 0.3, and 0.5) % of eggplant peel and calyx pectin at room temperature reached (28, 31.6, 35) % respectively, (Figure 4), and the rate of foam stability for the three concentrations registered (31.5) % (Table 3). The results

showed that the stability of the foam increased significantly with increasing the concentration of pectin, and the highest stability of the foam was found in eggplant peels and calyxes of 35% at a concentration of 0.5%. Pectin extracted from eggplant peels and calyxes is distinguished by the formation of foam and good stability because pectin extracted from eggplant residue contains multiple sugars, and the reason for the stability of the foam is due to its high viscosity, which helps to form a network that prevents air bubbles from binding to each other (18).

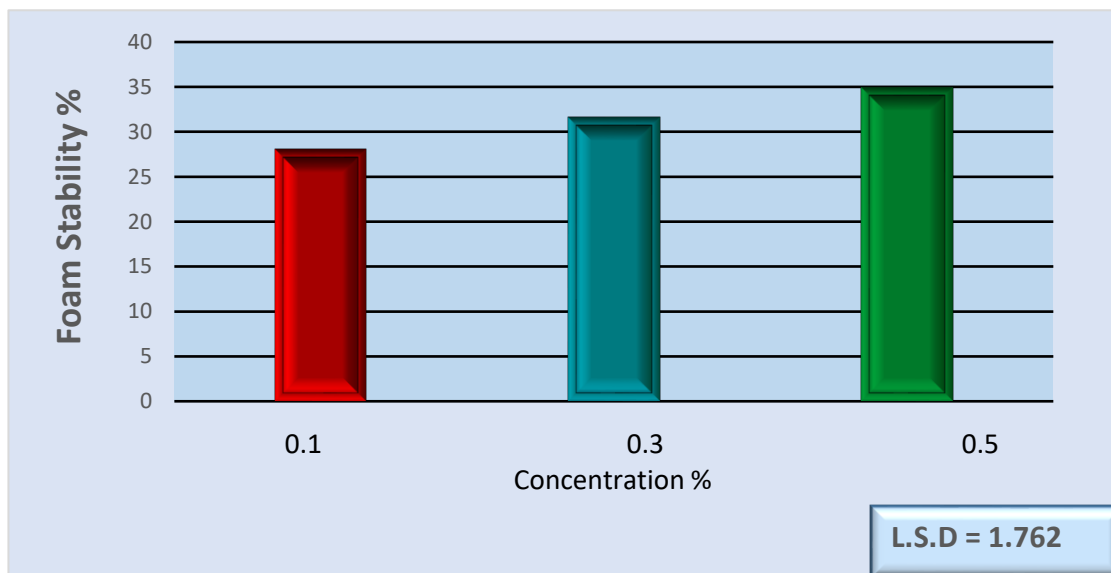


Figure (4). The relationship between the concentrations of pectin extracted from eggplant peels and calyxes with foam stability

Table(3). Functional properties of pectin extracted from eggplant peels and calyxes

Functional Features	Value
Water holding capacity (water(g)/pectin powder(g))	3.3
Fat holding capacity (fat(g)/pectin powder(g))	2.86
Emulsifying property (%)	75.8
Foaming property (%)	71.3
Foam stability (%)	31.5

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