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Optimization and Characterization of Pectin Hydrogel from Tamarind Pulp: FTIR, Viscosity and Yield Analysis

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ABSTRACT

Background and Objective: Tamarind (*Tamarindus indica*) is a tropical fruit known for its rich flavor and diverse applications across food, pharmaceutical and cosmetic industries. This study investigates the production and optimization of pectin hydrogel derived from tamarind pulp, focusing on its potential pharmaceutical applications, particularly in wound dressing. **Materials and Methods:** The primary variables tested were temperature, extraction time and glycerol concentration, which were adjusted to maximize the yield of pectin. The design allowed for the investigation of interactions between these factors, leading to the identification of optimal conditions for pectin extraction. The study setting was a controlled laboratory environment, where tamarind pulp was processed to extract pectin. Statistical analysis revealed a significant quadratic model with an R^2 value of 0.9633, indicating good fit and predictive power. **Results:** Fourier Transform Infrared Spectroscopy (FTIR) analysis confirmed the presence of functional groups such as hydroxyl (-OH), methylene (-CH2-) and amide (-CO-NH), which are essential for drug delivery, bioadhesion and biodegradability in pectin hydrogels. The viscosity of the produced pectin was found to be 300 cP at 25°C, suitable for hydrogel applications. The highest yield of 60% was achieved at 100°C, 15 min and 4 mL of glycerol. Validation experiments showed a yield of 59.60%, closely matching the theoretical yield with an error of -0.67%. **Conclusion:** These findings demonstrate the feasibility of large-scale production of pectin hydrogel from tamarind pulp, with potential for pharmaceutical applications.

KEYWORDS

Pectin, hydrogel, FTIR spectrum, viscosity, bioadhesion, tamarind pulp

INTRODUCTION

Tamarind (*Tamarindus indica*) is a tropical fruit known for its rich flavor and diverse applications across the food, pharmaceutical and cosmetic industries. The fruit's unique composition of polysaccharides, polyphenols and fatty acids endows it with valuable functional properties that impact its utilization[1-5].

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Proximate analysis of *Tamarindus indica* fruit pulp is essential for understanding their nutritional and functional characteristics, which significantly influence their potential applications in industrial products and other value-added goods[6-8].

Pectin, a natural polysaccharide primarily found in the cell walls of plants, particularly in fruits, has garnered significant attention for its versatile applications in the pharmaceutical, food and cosmetic industries[9-14]. Owing to its biocompatibility, non-toxicity and ability to form hydrogels, pectin is extensively utilized in drug delivery systems, wound dressings and as a stabilizer in various formulations[15-17].

Hydrogels are three-dimensional polymeric networks capable of holding large amounts of water, making them ideal for various biomedical applications, including wound dressings and drug delivery. The functional properties of pectin hydrogels, such as swelling behavior, bioadhesion and controlled release, are largely dependent on the presence of specific functional groups within the pectin structure[18,19]. Understanding these functional groups through analytical techniques like Fourier Transform Infrared Spectroscopy (FTIR) is crucial for optimizing the hydrogel's performance for specific applications.

This study focuses on the production, characterization and optimization of pectin hydrogels extracted from tamarind pulp. The FTIR analysis was employed to identify the functional groups present in the pectin hydrogel, while viscosity measurements were conducted to determine the suitability of the hydrogel for wound dressing applications[20-22]. The extraction process was optimized using a response surface methodology, considering factors such as temperature, extraction time and glycerol concentration to maximize the yield of pectin hydrogel. The optimized conditions were validated through laboratory experiments and the results were analyzed to ensure the reproducibility and scalability of the production process. The objective of this study was to investigate the production and optimization of pectin hydrogel derived from tamarind pulp, focusing on its potential pharmaceutical applications, particularly in wound dressing.

MATERIALS AND METHODS

Study area and sites: This study was conducted in Zaria, Kaduna State, Nigeria. It is located at 11.12°N Latitude and 7.73°E Longitude and it is situated at an elevation of 640 m above sea level. The population of Zaria is 766,000, making it one of the most populous cities in Kaduna State[23].

Sample collection and analysis: *Tamarindus indica* A, B and C (Fabaceae) were purchased from a local market in Kano, Kaduna and Katsina, respectively; tamarind fruit weighing 5 kg was boiled in distilled water at varying temperatures (50, 60, 70, 80, 90 and 100°C) for varying times (15, 30, 45 and 60 min) in which the temperature and time of each sample were recorded and tamarind pulp was produced. The produced pulp was mashed or stirred with a stirrer to create a homogeneous mixture and the homogenous mixture was strained or filtered through a cheesecloth or filter paper to separate the slurry from the residue and seeds and the seeds were discarded or utilized for another purpose and a slurry was produced, 100 mL of ethanol (95% v/v) with 5 mL of water) was added to the slurry and was stirred and allowed to settle for a considerable time in which a precipitated liquid was formed. The 0.5, 1, 2, 3 or 4mL of glycerol was added to the precipitated liquid and mixed well to create a homogeneous glycerol blend mixture, hence glycerol blind mixture was formed. The glycerol blend mixture was centrifuged at 3000 rpm for 10 min to separate it from the liquid and the liquid was discarded and the precipitate was retained. The retained precipitate was collected and preserved as pectin hydrogel and utilized for further analysis. This study spanned from January to November 2023[24,25].

Tools and equipment manufacturers: The equipment utilized in this study, including the viscometer, rheometer and various other tools, were sourced from manufacturers such as Hanna Instruments (Woonsocket, Rhode Island, USA), Thermo Fisher Scientific (Waltham, Massachusetts, USA) and Mettler Toledo (Columbus, Ohio, USA), among other reputable suppliers[26,27].

Determination of viscosity of pectin hydrogel: A known quantity of pectin hydrogel was prepared and poured into the viscometer's sample holder and was ensured to fill the recommended level, (specifically, rheometer) in which the gelation was induced through a pH adjustment. The viscometer was set to the desired temperature of 25°C, which is a standard temperature for viscosity measurements. The viscometer (rheometer) was started and was allowed to rotate at a constant speed and shear stress was applied to the sample. The viscometer measured the resulting shear stress which allowed the dynamic viscosity of pectin hydrogel to be determined. The operation was repeated severally to ensure accuracy and reliability of the result. The viscosity was found to be 300 cP (centipoise) at 25°C, indicating a relatively thick and viscous fluid. This value is important for understanding the flow behavior of the pectin extract, which is crucial for its potential applications in pharmaceuticals, most especially wound dressing or healing[28,29].

Statistical analysis: The statistical analysis was performed using the BMDP 2R program for stepwise multiple regression. Results were expressed as the mean of triplicate analyses[30,31]. The results of the viscosity analysis of pectin hydrogel were obtained at a significance level of $p<0.05$.

Optimization of pectin hydrogel: The details from Table 1 were used for the laboratory experiments strictly as scheduled. Based on the above procedure for the production of pectin each of the above steps is used for each detail in the table above and the yield for each detail is recorded as the response.

Table 2 above shows that the equation is quadratic which shows non-linearity.

Table 1: Experimental design for pectin hydrogel extraction from tamarind pulp

	Factor 1	Factor 2	Factor 3	Response 1
Run	A: Temperature (°C)	B: Time (min)	C: Glycerol (Ml)	Yield (%)
1	100	15	4	-
2	33	38	2.3	-
3	50	60	0.5	-
4	100	15	0.5	-
5	75	38	0.7	-
6	75	0.3	2.3	-
7	75	38	2.3	-
8	75	38	5.0	-
9	75	75	2.3	-
10	117	38	2.3	-
11	100	60	4	-
12	50	60	4	-
13	50	15	4	-
14	100	60	0.5	-
15	75	38	2.3	-
16	75	38	2.3	-
17	75	38	2.3	-
18	75	38	2.3	-
19	75	38	2.3	-
20	50	15	0.5	-

Table 2: Fit summary for pectin hydrogel yield optimization model

Source	Sequential p-value	Lack of fit p-value	Adjusted R^2	Predicted R^2
Linear	0.0195	0.0032	0.3488	0.0523
2FI	0.0228	0.0091	0.6058	0.0148
Quadratic	0.0001	0.3394	0.9303	0.7597 suggested
Cubic	0.3394		0.9437	Aliased

Table 3: Analysis of variance (ANOVA) for quadratic model result

Source	Sum of squares	df	Mean square	F-value	p-value	
Model	3901.54	9	433.50	29.16	<0.0001	Significant
A	993.02	1	993.02	66.80	<0.0001	
B	177.44	1	177.44	11.94	0.0062	
C	303.88	1	303.88	20.44	0.0011	
AB	162.00	1	162.00	10.90	0.0080	
AC	84.50	1	84.50	5.68	0.0383	
BC	882.00	1	882.00	59.33	<0.0001	
A^2	376.46	1	376.46	25.32	0.0005	
B^2	35.73	1	35.73	2.40	0.1521	
C^2	522.76	1	522.76	35.16	0.0001	
Residual	148.66	10	14.87			
Lack of fit	88.66	5	17.73	1.48	0.3394	Not significant
Pure error	60.00	5	12.00			
Cor total	4050.20	19				

A: Temperature, B: Time and C: Glycerol

Table 4: Statistical summary and R-squared values for pectin hydrogel yield optimization

Parameter	Values
Std. Dev.	3.86
Mean	35.30
CV (%)	10.92
R^2	0.9633
Adjusted R^2	0.9303
Predicted R^2	0.7597
Adeq precision	14.6545

The Final Equation in Terms of Actual Factors as cited by Imoisi *et al.*[25]:

$$Pectin(\%)=26.03+8.53*A+3.62*B+5.45*C+4.50*AB+3.2*AC-10.50*BC+5.09*A^2+1.56*B^2+7.73*C^2$$

Where

A = Temperature

B = Time

C = Glycerol

In Table 3, the model F-value and of 29.16 implies the model is significant p-values and less than 0.0500 indicate model terms are significant. In this case, A, B, C, AB, AC, BC, A^2 and C^2 are significant model terms and values greater than 0.1000 indicate the model terms are not significant. The Lack of Fit F-value and of 1.48 implies the Lack of Fit is not significant because its probability greater than the f-value is 0.3394. From the Table 3 it can be seen that the model is significant and consistent and good for prediction. Also, ANOVA indicates the effect of independent variables on dependent variables.

The R^2 indicates the goodness and the fitness of the model, while predicted R^2 signifies the predicting power of the model, and adjusted R^2 estimates the predictive performance of the model new data. The R^2 value 0.9633 close to 1 indicates that the model is good and fit as shown in Table 4. The Predicted R^2 of 0.7597 is in reasonable agreement with the Adjusted R^2 of 0.9303; i.e. the difference is less than 0.2. Adequate Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 14.654 indicates an adequate signal. This model can be used to navigate the design space.

RESULTS AND DISCUSSION

Based on the FTIR spectrum, the band peaks at 3268cm^{-1} indicating the presence of hydroxyl (-OH) groups, which are abundant in pectin's galacturonic acid and rhamnose units. The 2885cm^{-1} suggests the presence of methylene (-CH2-) group, which is present in pectin's backbone, 1330cm^{-1} indicates the presence of amide (CO-NH) group which may indicate pectin's interaction with amino acid or protein,

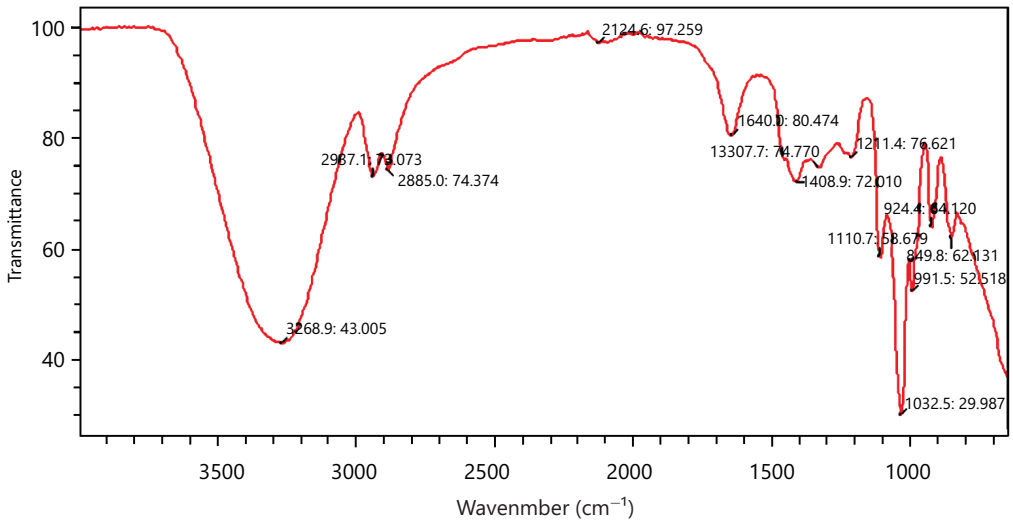

Fig. 1: FTIR spectrum of pectin extracted from tamarind pulp

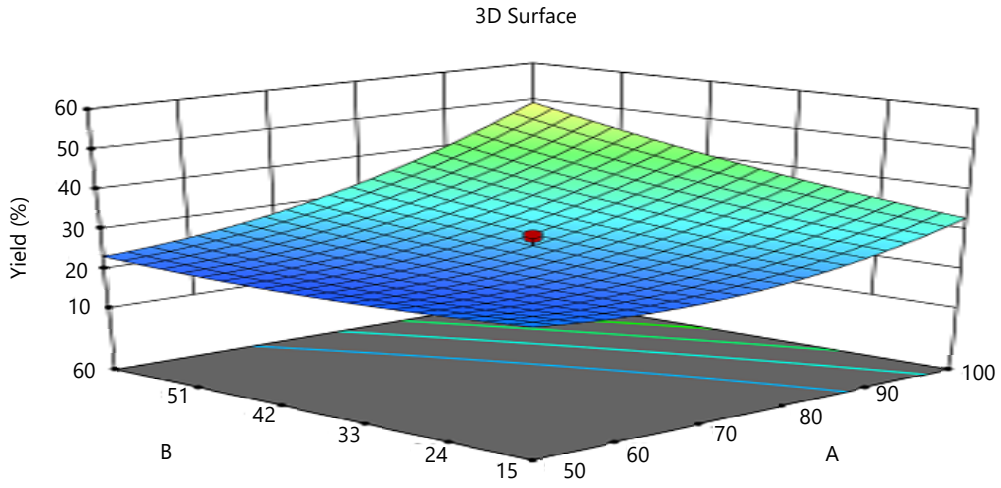

Fig. 2: Contribution of time and temperature to extraction yield
A: Temperature (°C) and B: Time (min)

1408 cm^{-1} suggest the presence of methyl (-CH3-) group which are present in pectin's rhamnose, 1110 cm^{-1} indicate the presence of either (C-O-C) group which are abundant in pectin's backbone., 1032 cm^{-1} suggest the presence of hydroxyl (-OH-) group and / or ether (C-O-C) group, which are abundant.

Based on the spectrum the functional groups present in pectin hydrogel are hydroxyl (-OH), methylene (-CH2-) group, amide (-CO-NH), methyl (-CH3-) groups and ether (C-O-C) groups as shown in Fig. 1. In pharmaceutical applications, these functional groups enable pectin hydrogels to control drug release and absorption, enhance bioadhesion and tissue interaction, provide a biocompatible and biodegradable matrix, offer pH-dependent swelling and release properties and enable cross-linking and gelation for tailored properties etc.

The FTIR spectrum and Table 5 showed: 3268 cm^{-1}: This peak corresponds to hydroxyl (-OH) groups, which are abundant in the galacturonic acid and rhamnose units of pectin. These hydroxyl groups play a crucial role in the gelation process and contribute to the hydrophilicity of pectin, making it suitable for hydrogel formation, 2885 cm^{-1}: The presence of the methylene (-CH-) group suggests the presence of aliphatic chains in the pectin backbone. These groups are essential for the structural integrity and viscosity

Table 5: Functional groups identified in pectin extracted from tamarind pulp

Name	Chemical symbol/group	Peak bank (cm^{-1})
Hydroxyl	(-OH)	3268
Methylene	(-CH2-)	2885
Ester or carboxyl	(CO-O-R)/(COOH)	1330
Ether	(C-O-C)	1408
Carboxylate	(COO)	1110

Table 6: Viscosity ranges of pectin for various applications

Application	Range (Cp or mPs.a)
Pharmaceutical	100-10,000
Jam and jellies	500-5000
Coating film	1000-10,000
Stabilizer	1000-5000
Bakery products	500-2000
Dairy products	1000-5000

Table 7: Constraints and goals for pectin extraction optimization

Name	Goal	Lower limit	Upper limit	Lower weight	Upper weight	Importance
A	Goal is in range	50	100	1	1	3
B	Goal is in range	15	60	1	1	3
C	Goal is in range	0.5	4	1	1	3
Yield	maximize	20	60	1	1	3

A: Temperature, B: Time and C: Glycerol

Table 8: Optimal solutions for pectin extraction

A temperature	Time	Glycerol	Yield	Desirability	
100.000	15.000	4.000	60.053	1.000	Selected
99.546	15.000	4.000	59.737	0.993	
100.000	15.047	3.984	59.725	0.993	
100.000	17.172	4.000	59.531	0.988	
99.979	15.000	3.968	59.406	0.985	
100.000	18.743	4.000	59.171	0.979	
99.931	15.000	3.949	59.008	0.975	
100.000	19.812	4.000	58.935	0.973	
100.000	60.000	0.500	58.891	0.972	
99.783	60.000	0.500	58.718	0.968	

of the hydrogel and 1330 cm^{-1}: This peak indicates the presence of an ester/carboxyl (CO-O-R)/(COOH) group, which may arise from interactions between pectin and proteins or amino acids[32-37]. Such interactions could influence the bioadhesion properties of pectin hydrogels, making them useful in tissue engineering and drug delivery applications. The 1408 cm^{-1}: Presence of an ether (C-O-C) group, particularly in the rhamnose moieties, may influence the hydrophobic interactions within the hydrogel network, affecting its mechanical properties and stability and 1110 cm^{-1}: These peaks are associated with carboxylate (COO) group, indicating the complex polysaccharide structure of pectin. The ether groups contribute to the flexibility and gelation properties of the hydrogel, while the hydroxyl groups further enhance water absorption and retention[38,39]. The functional groups identified are consistent with pectin's ability to form hydrogels that are biocompatible, biodegradable and capable of controlled drug release, making them ideal for pharmaceutical applications such as wound dressings.

The viscosity of the pectin produced was found to 300 centipoise (cP) at temperature of 25°C which is suitable for wound dressing construction as shown in Table 6. The viscosity range is typically considered appropriate for creating hydrogel-like material that can provide a protective barrier absorbed excess fluid and promote wound healing.

As shown in Fig. 2, the temperature has more contribution to extraction yield than time because at 100°C and a time of 15 min highest yield was achieved. At a point the time was somewhat not relevant to the extraction as you move on the time axis that temperature axis this signifies that temperature has more impact that time since glycerol has been employed to improve its quality.

Table 7 shows that the entire variables are constrained to be within the range and that the goal is to maximize the yield.

From as shown in Table 8 above 69 solutions were found as optimization result but only few are selected here in which No 1 was tested in the laboratory to validate the laboratory experiment which initially gave a yield of 60% as highest at temperature of 100°C, time of 15 min and 4 mL of glycerol.

The validation experiment conducted under the optimized conditions (temperature of 100°C, time of 15 min and 4 mL of glycerol) resulted in a pectin hydrogel yield of 59.60%, which is in excellent agreement with the laboratory experiment yield of 60%. This confirms the reproducibility and scalability of the optimized conditions, demonstrating the reliability of the process for large-scale production of pectin hydrogel from tamarind pulp[25]:

$$\text{Error} = \frac{\text{Experimental value} - \text{Theoretical value}}{\text{Therorectical value}} \times 100$$

Where
Experimental value = 59.60%
Theoretical value = 60% (laboratory experiment yield)

$$\text{Error} = \frac{59.60 - 60}{60} \times 100$$

$$\text{Error} = \frac{-0.40}{60} \times 100$$

$$\text{Error} = -0.67\%$$

The error is -0.67%, which means that the experimental value is 0.67% lower than the theoretical value. The negative sign indicates that the experimental value is lower than the theoretical value. The Fourier-transform Infrared Spectroscopy (FTIR) analysis of pectin revealed key functional groups that are essential to its chemical structure and its applications, particularly in the pharmaceutical field. The viscosity of the pectin hydrogel was measured at 25°C, yielding a value of 300 cP. This viscosity falls within the range suitable for wound dressing construction, where the hydrogel must provide a protective barrier, absorb excess fluids and promote wound healing. The viscosity was determined using a rheometer, which applied shear stress to the sample, allowing for the calculation of dynamic viscosity. The consistency of the measurements across multiple trials confirms the reliability of the pectin hydrogel's flow behavior, which is crucial for its application in biomedical fields[40-44].

The temperature and time variations were crucial in optimizing the yield. Higher temperatures generally increased the yield, as seen with the highest yield of 60% obtained at 100°C and 15 min. Homogenization and filtration were essential to produce a smooth slurry, necessary for efficient precipitation and pectin recovery[45,46]. The addition of ethanol facilitated the precipitation of pectin from the slurry, with the quantity of glycerol added later influencing the final hydrogel properties. This step ensured the separation of the hydrogel from the liquid, concentrating the pectin for further use[47,48].

The optimization process used a design of experiments (DoE) approach, focusing on temperature, time and glycerol concentration as independent variables. The highest yield was 60% at 100°C, 15 min and 4 mL of glycerol, highlighting the significant impact of temperature on pectin extraction. The quadratic model derived from the ANOVA results showed high significance, with a model F-value of 29.16 and an

R^2 value of 0.9633, indicating a good fit. The predicted and adjusted R^2 values were in close agreement, further confirming the model's reliability. The perturbation plot and 3D surface plots emphasized that temperature had a more substantial effect on yield than time and the interaction between variables was critical in optimizing the process. The validation experiment, conducted under the optimized conditions, yielded 59.60% pectin hydrogel, which closely matched the theoretical yield of 60%, with a minor error of -0.67%. This small discrepancy indicates that the experimental process is robust and scalable for industrial production. The study successfully characterized and optimized the production of pectin hydrogel from tamarind pulp, demonstrating its potential for pharmaceutical applications. The combination of FTIR analysis, viscosity measurement and optimization through DoE provided comprehensive insights into the properties and production of pectin hydrogel, paving the way for its use in wound healing and drug delivery systems[49,50].

CONCLUSION AND RECOMMENDATIONS

The FTIR analysis identified essential functional groups-hydroxyl, methylene, amide, methyl and ether-that enhance the hydrogel's pharmaceutical potential, particularly for drug delivery and wound dressing. The measured viscosity aligns well with medical application requirements, making the hydrogel suitable for wound care. The quadratic model demonstrated robust predictive accuracy, confirming the optimization's effectiveness. Future research should explore scaling the production process and investigating the hydrogel's performance in various medical applications to fully leverage its potential.

SIGNIFICANCE STATEMENT

This study explores the extraction and characterization of pectin hydrogel from tamarind pulp, aiming to evaluate its potential for pharmaceutical applications, particularly in wound dressing. The research successfully optimized the extraction process, achieving a high pectin yield with an error margin of only -0.67%, which underscores the method's precision and scalability. Key functional groups were identified through FTIR analysis, confirming the hydrogel's chemical composition and its suitability for medical use. The study also identified optimal viscosity (300 cP at 25°C), which is critical for the desired application in wound dressings. The findings suggest that tamarind-derived pectin could be a promising material for future development in the biomedical field, particularly for products requiring biocompatible and effective hydrogels.

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