Synthesis and Characterization of Pectin Membrane as a Matrix for Curcumin Sustained-Release

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Abstract. This study aims to synthesize pectin membranes, characterization of pectin membranes use FTIR, XRD, SEM, and to see the loading of curcumin in pectin membranes as a drug delivery system by contact time variations. Pectin membrane synthesize by dissolved 0.5 gram of pectin powder in 25 ml of 2% acetic acid solution then stirred for 1 hour. The solution is molded in membrane mold then oven for 24 hours at 50°C. Pectin membranes were characterized by FTIR to determine functional groups, XRD test to determine crystallinity, and SEM test to determine the surface morphology. The loading of curcumin in pectin membrane are carried out by contacting the pectin membrane into 100 ppm curcumin at various times of 5 to 120 minutes. Determination of curcumin concentration contained in pectin membrane use UV-Vis spectrophotometer at λ 426 nm. The results showed that the produced of pectin membrane was thin, pellucid, and homogeneous sheets. The results of FTIR characterization showed OH group at wave number 3448.72 cm\(^{-1}\) and carbonyl group at wave number 1604.77 cm\(^{-1}\). XRD characterization result showed that the pectin membrane was amorf. SEM characterization results showed smooth and flat surface morphology. The optimum contact time in loading curcumin process in pectin membrane was 60 minutes with a loading percentage of 10%.

1. Introduction

Curcumin is a compound extract from Curcuma Longa. It is often found in Indonesia commonly called turmeric which is used for herbs and herbal medicines. Curcumin contains bioactive compounds that are potentially used as drugs. Many studies have suggested various benefits of curcumin, including anti-cancer drugs, anti-inflammatory, antioxidant, antibacterial, antineoplastic, antiproliferative, etc. [1]. However, curcumin has several disadvantages including poor solubility and absorption, rapid metabolism, low bioavailability, etc. [2]. Therefore, it needs a matrix that can load curcumin and then release it according to the absorption target in order to function optimally.

Sustained-release designed to release a drug in the body slowly over an extended period of time [3]. Moreover, sustained-release is designed to release a therapeutic dose proper initial drug (loading dose) followed by drug release constant and slower. The speed of drug release is designed in such a way that the amount of medicine lost from the body due to the elimination is constantly replaced. The advantage is drug spread evenly into the blood without repeated dosing.

Pectin is a complex polysaccharide that has a main component of esterified D-galacturonic acid. Pectin is the main constituent of the cell wall of higher plants [4]. Pectin is not soluble in ethanol or other organic solvents, but it is soluble in water. The use of pectin as a matrix drug sustained release is...
based on its ability to form a gel. Pectin gel formation is caused by hydrogen bonds between hydroxyl groups around molecules and between carboxyl groups of pectin molecules [5]. Otherwise, pectin is a biopolymer which still exists in the digestive system and then degraded by the pectinolytic enzymes in the colon. So that pectin is suitable as a drug delivery matrix that targets the colon [6]. This research aims to synthesize pectin membranes, characterization of pectin membranes use FTIR, XRD, SEM, and to see the loading of curcumin in pectin membranes as a drug delivery system by contact time variations.

2. Materials and method

2.1. Materials
Pectin powder, Acetic Acid 2% (Mallincord), Ethanol 70% (Mallincord), Curcumin (Merck), and Distilled water.

2.2. Method

2.2.1. Synthesis of Pectin Membrane. 0.5-gram pectin powder dissolve in 25 ml acetic acid 2% then stirred for an hour till homogeneous. Gel pectin printed in polypropylene container then oven at 50°C for 24 hours to form pectin membrane. After that, pectin membrane characterized using FTIR, XRD, and SEM.

2.2.2. The Loading of Curcumin in Pectin Membrane. 50 mg of pectin membrane was immersed in a 25 ml curcumin solution 100 ppm then stirred. The percentage of loading was observed in time intervals of 5, 10, 15, 30, 45, 60, and 120 minutes. The concentration of curcumin loaded on pectin membrane was measured by UV Vis Spectrophotometer at λ 426 nm.

3. Results and discussion
Pectin membranes are prepared by dissolving pectin powder in 2% acetic acid solution without heating, then printed in a polypropylene container and dried with an oven. Drying in the oven aims to evaporate the solvent, acetic acid, so the remains is pectin in the forming of water free membrane. The pectin membrane produced is clear, flexible and not easily torn as shown in Figure 1a.

The procedures of sustained materials or drug sustained release is focused on the plate (film) model. The loading of curcumin into the pectin membrane by submersion 100 ppm curcumin solution in 70% ethanol into the pectin membrane for 2 hours. The dissolution of curcumin in 70% ethanol has the reason that curcumin can dissolve completely in 70% ethanol. Percentage of loading was observed in time intervals of 5, 10, 15, 30, 45, 60, and 120 minutes. The loading has the purpose of trapping curcumin into the pore of the delivered material and store it until the releasing process is carried out. In this study curcumin contained in the pectin membrane was yellow, thin, flexible and not easily torn that was shown in Figure 1b.

3.1. FTIR Characterization
FTIR characterization was used to know the functional group contained in the pectin membrane. The results of FTIR characterization showed OH group at wave number 3448.72 cm⁻¹ and carbonyl group at wave number 1604.77 cm⁻¹. Pectin spectrum has typical absorption of a strong band between 1630-1600 cm⁻¹ which is a stretching band of carboxylic ions. The wave number 1300 cm⁻¹ showed COO-stretching group. The result of pectin membrane showed in Figure 2.
Figure 1. a) pectin membrane, b) Pectin membrane loaded curcumin

Figure 2. FTIR Characterization of Pectin Membrane

3.2. XRD Characterization

Pectin membrane was also analyzed in terms of crystallinity using XRD characterization. The thermogram showed that pectin membrane is semicrystalline that indicated by high peak intensity with angles 2θ at 22°. It caused that pectin membrane can load the curcumin.

3.3. SEM Characterization

Pectin membrane was analyzed by SEM characterizations in order to see the morphology of surface area which is showed in Figure 4. The morphology of pectin powder showed rough structure that distributed in the particle while membrane showed microporous smooth membrane. So pectin membrane has stable structure than pectin powder.
Figure 3. XRD Characterization of Pectin Membrane

Figure 4. SEM Characterization of pectin membrane and pectin powder
3.4. Encapsulation of Curcumin

The process of loading curcumin into the pectin membrane aims to trap curcumin into the pore membrane pectin with an adsorption model. The loading time determines the optimum time of drug compounds restrained in the pore of the conductor, so that certain concentrations or masses of the drug can be found optimum in the conductor material. In this research curcumin loading by submersion 100 ppm curcumin in 70% ethanol for 2 hours. The optimum time for loading curcumin into the pectin membrane was 60 minutes with curcumin loaded at 10% shown in Figure 5.

![Graph of Loading Curcumin into Pectin Membrane](image)

**Figure 5.** Graph of Loading Curcumin into Pectin Membrane

Calculation of the percentage efficiency of encapsulation using the formula:

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\%EE = \left( \frac{[\text{Curcumin}]_{\text{total}} - [\text{Curcumin }\text{loaded}]}{[\text{curcumin }\text{total}]} \right) \times 100\%.
\] (1)

The optimum time for loading of curcumin into the pectin membrane is 60 minutes with the percentage efficiency of encapsulation is 10%. The efficiency pattern of the curcumin loading over time showed that curcumin was initially loaded gradually increased into the membrane. The loading of curcumin decreased after 60 minutes with a percentage efficiency of encapsulation is 10%. That proves the loading of curcumin by pectin membrane has been saturated.

4. Conclusion

The Pectin membrane has been successfully synthesized based on the result of FTIR, XRD, and SEM characterizations. The results of FTIR characterization showed that pectin membrane has an active functional group. There are carbonyl groups at wave number 1604.77 cm\(^{-1}\) and hydroxyl groups at wave number 3448.72 cm\(^{-1}\). Based on XRD characterization pectin membrane was semi-crystalline and based on SEM characterization, the pectin membrane showed smooth and flat surface morphology. Curcumin can be loaded into pectin membrane with an optimum time of 60 minutes. The percentage efficiency of encapsulation is 10%.

Acknowledgment

The authors express gratitude to the Indonesian Ministry of Research and Technology Directorate of Higher Education for fundamental research grant and Universitas Sebelas Maret for the facility.

References

[1] Prasong S, Thidarat C, Naruemon K, Maneerat C and Duangporn W 2019 *J.Heliyon*. 5 e02222
[2] Mousumi K, Pritam S, Novel G, Sharmistha C, Prasenjit M, Joydeep D and Parames C 2019 *J. Advanced Research*. 18 162-172
[3] Susana R, Joyce T and Pedraza C 2018 J.Nutrition & Intermediary Metabolism. 14 29-41
[4] Purwaningsih E 2016 J.Kedokteran Yarsi. 24 203-211
[5] Hashem A, Mahnaz T, Mohammadifarc A and Hamishehkar H 2017 J.Biological Macromolecules. 97 16–22
[6] Lena N and Havazelet B 2017 J.Biological Macromolecules. 101 852–861