IJNRD.ORG ISSN : 2456-4184
INTERNATIONAL JOURNAL OF NOVEL RESEARCH AND DEVELOPMENT (IJNRD) | IJNRD.ORG
An International Open Access, Peer-reviewed, Refereed Journal

STUDIES ON MECHANICAL, BARRIER AND ANTIMICROBIAL PROPERTIES OF BIODEGRADABLE FILMS PREPARED USING STARCH AND GLYCEROL

Kavinya S[1], Harini E S[2], Ranjith Kumar S[3], Deepika R M[4]

[1]Assistant professor, [2]Student, [3]Student, [4]Assistant Professor

Department of Food Technology

Kongu Engineering College, Perundurai, Erode, India

Abstract: In the current research work biodegradable films were prepared using corn starch (0-2%), agar (1- 3%), and glycerol (0.5-1.5%). Three levels Box Behnken Design (BBD) under Response Surface Methodology (RSM) were employed to study the influence of variables on the mechanical, barrier, and biodegradable properties. The casting method was followed to produce transparent films. The results showed that the addition of glycerol decreases the modulus elasticity and tensile strength of the films. The results were analyzed by Pareto analysis of variance (ANOVA) and a second-order polynomial equation was developed for all responses to predict the effects of variables on responses. The optimized film was found at 2.91% of agar, 1.39% of starch, and 0.5% of glycerol. Anti-microbial properties of garlic extract were studied for the optimized film against E.Coli microorganisms.

Keywords: Biodegradable films, mechanical properties, barrier properties, biodegradable properties, RSM, ANOVA.

1. INTRODUCTION:

Environmental pollution is a critical issue, with plastic pollution being a significant contributor. Plastics, defined as moldable and hardenable man-made organic materials, include resins, resinoids, and polymers. Approximately 40% (161 million tons) of the annually produced plastic is used for packaging and is often discarded after a single use, resulting in 18 billion pounds of plastic waste each year. This waste accumulates on land and in oceans due to its long decomposition time. In the food industry, plastics are crucial for safe storage and transportation, primarily in the form of rigid and flexible plastics. However, researchers are exploring alternatives to flexible plastics, such as films made from renewable resources like starch. Starch, a low-cost, degradable, thermoplastic, is favored for bioplastic development due to its ability to form a continuous matrix. Starch-based films, which include polyesters for better processability and resistance, are typically made from corn starch, containing 27% amylose and the remainder amylopectin. Amylose aids in film formation through hydrogen bonding, while amylopectin can disrupt the polymer structure, leading to brittle films. Agar, a carbohydrate from marine algae, is also used to develop films, either foamed or combined with milk protein isolate. To improve flexibility and reduce brittleness in starch-based films, plasticizers like glycerol are added. Glycerol, with its small molecular size and multiple -OH groups, inserts between polymer chains, enhancing flexibility and transparency by reducing intermolecular forces. Vinegar, a 6% acetic acid solution, helps form homogenous films by hydrolyzing starch molecules. It increases starch solubility by releasing hydrogen and acetate ions, which react with and disorder the starch polymers, facilitating amylose structuring and improving film homogeneity. Vinegar also possesses antimicrobial properties, making it beneficial for food safety. Food safety is a major concern, particularly controlling microbial growth on food surfaces, a primary contamination source. Traditional methods, such as preservatives and surface treatments, are being reconsidered due to their negative health impacts. Current research focuses on natural antimicrobial agents like nisin, lysozyme, and plant extracts. Garlic, for instance, has high antimicrobial properties due to allicin, a molecule formed from alliin through the action of the enzyme alliinase. Allicin is effective against various microbes, including E. coli and Aspergillus species.

Overall, the shift towards natural, biodegradable materials like starch and agar for food packaging, enhanced by natural antimicrobial agents, addresses both environmental pollution and food safety concerns.

1.1 OBJECTIVES:

The objectives of the projects are

- To develop a biodegradable film and to evaluate the effects of variables such as starch, and glycerol on mechanical (tensile strength, modulus of elasticity, biodegradability).
- To evaluate the barrier properties (water vapor permeability) for the developed film.
- To evaluate the solubility characteristics of the developed film.
- To evaluate the antimicrobial properties of the developed film.

2. MATERIALS AND METHODS:

2.1. RAW MATERIALS

The raw materials such as corn starch, agar, glycerol and vinegar were used for production of biodegradable films. The glasswares and apparatus are Beakers, Pipettes, Petri plates, Crucible, Stirrer, Bowl, and Spatula, Prestige induction stove, laminar air flow chamber.

2.2. PREPARATION OF BIODEGRADABLE FILM

The film was prepared by casting method. The ingredients were optimized by RSM method which can be used for the preparation of this biodegradable film were 1.39% of corn starch, 2.91% of agar and 0.5% of glycerol. As per the design matrix ingredients of various quantities were mixed well and then it is heated at 65°C to 70°C for 15-20 minutes until the mixture turned transparent. It was then poured in petri dish on a leveled surface. Films were dried at room temperature for about 24 hours. Films were carefully removed from the petri dish and several tests were conducted using these samples to determine the mechanical and barrier properties of the films. Fig. 2.1 illustrates the flowchart for production of film.

Table 2.1 Preparation of biodegradable film

BIODEGRADABLE FILM	CORN STARCH (%)	AGAR (%)	GLYCEROL (%)
BF1	2	3	1.0
BF2	1	2	1.0
BF3	0	3	1.0
BF4	1	3	0.5
BF5	0	1	1.0
BF6	2	2	0.5
BF7	1	2	1.0
BF8	1	2	1.0
BF9	0	2	0.5
BF10	0	2	1.5
BF11	1	2	1.0
BF12	1	1	0.5
BF13	2	2	1.5
BF14	1	1	1.5
BF15	2	1	1.0
BF16	1	2	1.0
BF17	1	3	1.5

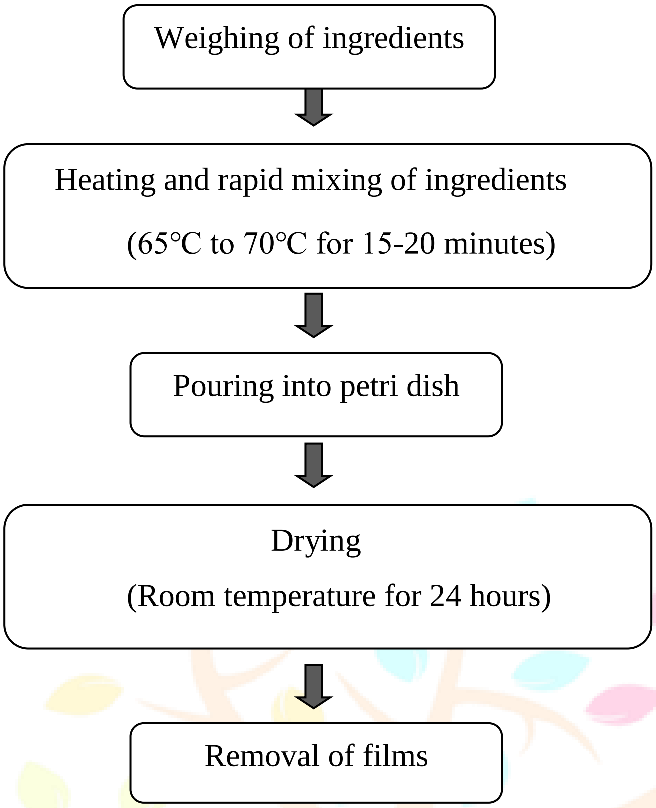

Fig. 2.1 Preparation of film

2.3. PREPARATION OF ANTIMICROBIAL FILM

To the optimized film composition by RSM, garlic extract was added to produce antimicrobial film. The film was optimized by setting goals for each response and desired condition. The preparation of this antimicrobial film 1.39% of corn starch, 2.91% of agar and 0.5% of glycerol. Garlic extract was prepared by taking garlic (8-12g) and was cut into small pieces. It was then boiled with 100ml water at 100° C for 15min. This extract was then mixed with starch, agar, glycerol and vinegar. Then the mixture was heated at 65-70°C for 15-20 minutes until the mixture turned transparent. It was then poured in petridish on leveled surface. Film was dried at room temperature for about 24 hours. Films were carefully removed from the petri dish and several tests were conducted using these samples to determine the mechanical, barrier and antimicrobial properties of the film. Production of antimicrobial film was as shown in Fig 2.3.

Table 2.2 Preparation of Antimicrobial film

Concentration of garlic extract (%)
8
10
12

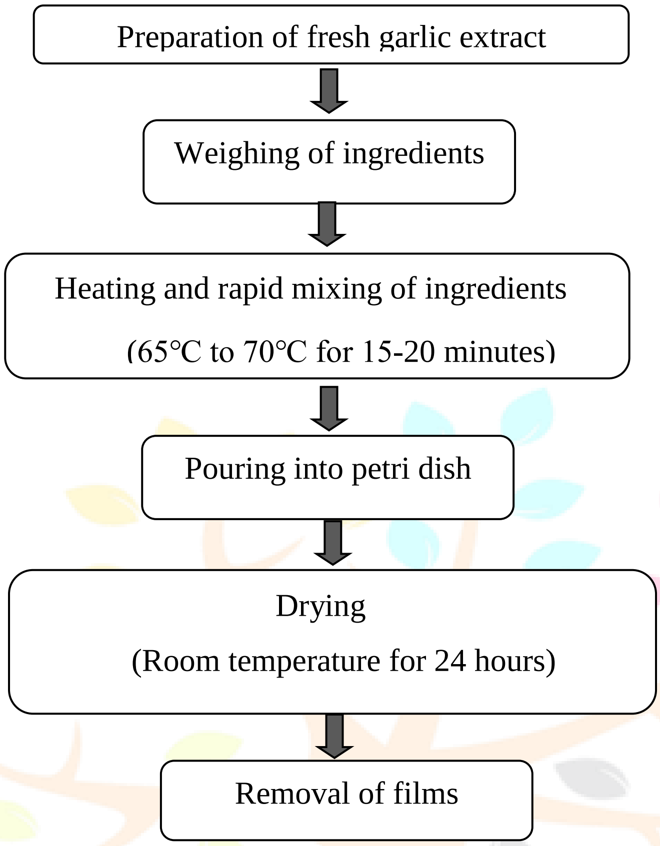

Fig. 2.3 Preparation of antimicrobial film

Fig. 2.3 Film preparation by casting

3. RESULTS AND DISCUSSION

In this chapter results and discussion, It deals with various parameters such as mechanical properties (tensile strength, Modulus of elasticity). Barrier properties (water vapour permeability, water solubility) and bio degradability. Response surface methodology (RSM) was used in the present work to develop the optimized composition for the three process variables: com starch, agar and glycerol were studied during experimentation.

3.1 DESIGN MATRIX

Based on Box Behnken Design (BBD), 17 runs with five replications was obtained, the design matrix for Mechanical properties and barrier properties were shown in the Table 4.1.

Table 3.1 Responses for design matrix

Run	Agar (%)	Starch (%)	Glycerol (%)	Tensile Strength (N/m)	Elasticity (N/m^2)	Water Permeability (g/m^2.day)	Solubility (%)	Biodegradability (%)
1	3	2	1.0	883.75	134806.20	65.13	32.73	67.70
2	2	1	1.0	774.85	65158.84	74.35	57.33	57.03
3	3	0	1.0	1222.50	113404.48	81.91	42.86	69.79
4	3	1	0.5	1360.25	347003.50	57.02	37.50	39.62
5	1	0	1.0	156.75	9029.35	106.58	78.32	39.88
6	2	2	0.5	905.75	193123.73	47.12	34.96	56.73
7	2	1	1.0	706.00	45431.13	57.02	53.90	64.70
8	2	1	1.0	782.32	50184.47	57.76	52.50	59.42
9	2	0	0.5	622.50	62187.83	43.07	71.05	37.74
10	2	0	1.5	605.00	55402.93	64.46	71.43	37.35
11	2	1	1.0	1289.25	102321.43	58.25	61.36	56.64
12	1	1	0.5	674.75	55672.43	72.99	92.50	59.20
13	2	2	1.5	655.75	32302.97	39.45	38.59	59.84
14	1	1	1.5	195.50	9873.73	98.23	62.50	57.12
15	1	2	1.0	363.25	19508.61	68.97	57.69	88.03
16	2	1	1.0	846.50	79038.30	64.19	58.33	55.19
17	3	1	1.5	1095.00	50460.65	51.43	57.41	45.20

3.2. Effect of Composition on Tensile Strength

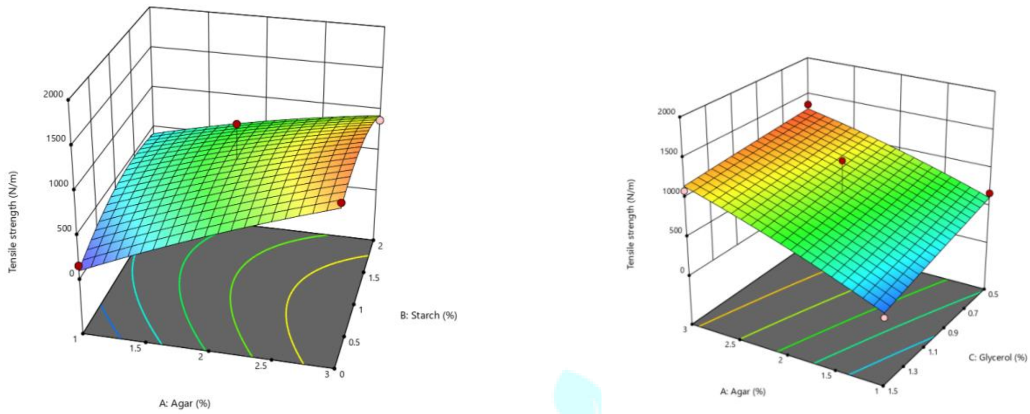

Fig. 3.1 Effect of composition on tensile strength

Effect of concentration on tensile strength was depicted on Fig. 3.1. From the figure it was inferred that when there is increase in agar and starch content, tensile strength increases due to the formation of intermolecular hydrogen bonds. With increase in glycerol content the tensile strength decreases due to more molecular mobility between polysaccharide chains which increases the plastic property. This increase in plastic effect leads to increase in free volume between the chains and it decreasing the tensile strength. Similar change in tensile strength with change in polyol concentration had been reported for films from potato starch.

3.3 Effect of Composition on Modulus of Elasticity

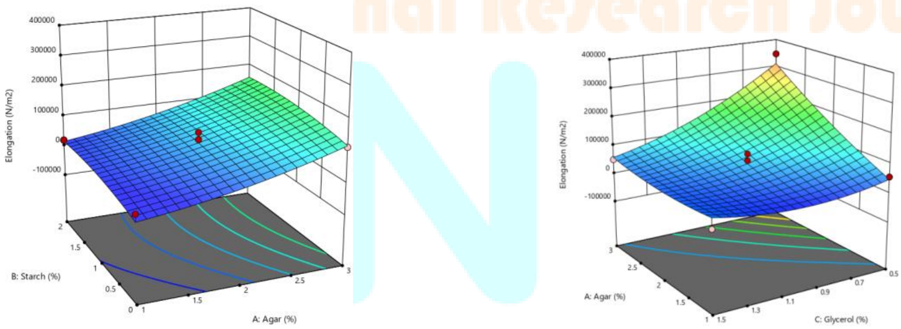

Fig. 3.2 Effect of composition on modulus of elasticity

The observed change in modulus of elasticity with different combinations of ingredients were shown in Fig 3.2. When the increase in agar and starch concentrations, the modulus of elasticity increases due to formation of hydrogen bonds between agar and starch. With increase in glycerol concentration the modulus of elasticity decreases reported similar observations that increase in glycerol content decreases in modulus of elasticity.

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3.4. Effect of Composition on Water Vapour Permeability

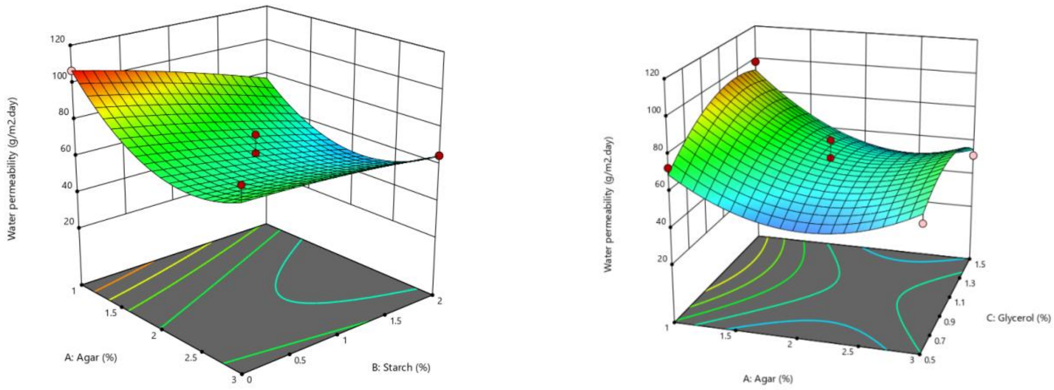

Fig. 3.3 Effect of composition on water vapour permeability

Fig 3.3 illustrated that increase in starch and agar decreases the water vapour permeability (WVP) of the films. Agar and starch cause formation of intermolecular interactions which decreases the free volume available for permeation of water vapour. Addition of glycerol increases the WVP by reducing the intermolecular bonds between polymer chains and thus increasing molecular space.

3.5. Effect of Composition on Solubility

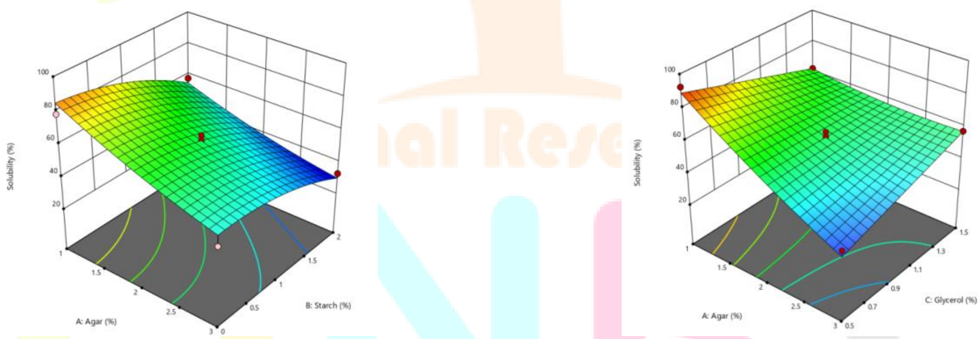

Fig. 3.4 Effect of composition on solubility

Effect of composition on solubility of the film was shown in Fig. 3.4. From the figure it was inferred that the solubility decreases with increase in concentration of starch, and agar. Starch contains amylose and amylopectin which tend to swell under the influence of water which decreases the solubility of film. Solubility increases with glycerol content because of the presence of aliphatic groups which changes the film structure which increases the solubility of the film.

3.6 Effect of Composition on Biodegradability

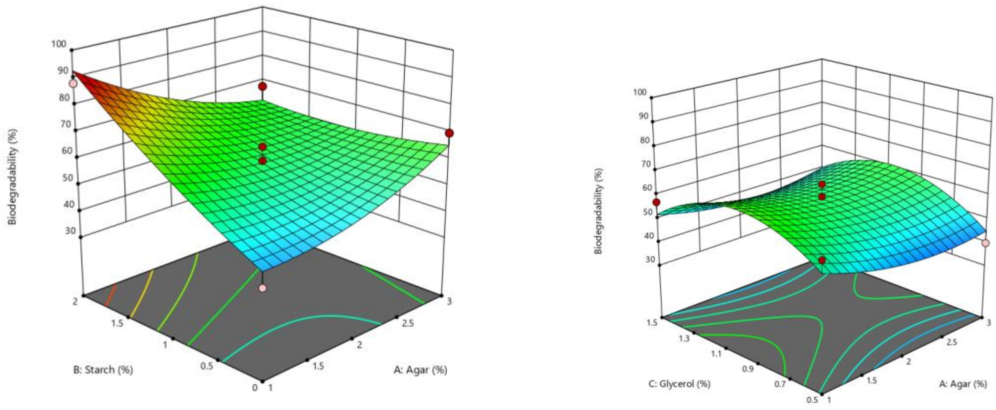

Fig. 3.5 Effect of composition on biodegradability

Fig 3.5 illustrated that increase in starch, agar and glycerol increases the biodegradability of the films. Starch and agar are hydrophilic in nature which absorbs water and tends to increase in water activity of the film. This leads to increase in the microbial activity on the film leading to weight loss. The glycerol in the films would be absorbed by soil which further increases the weight loss.

4.SUMMARY AND CONCLUSION

The production of biodegradable packaging is of great interest due to the ban of plastic based packaging materials. Corn starch and agar were chosen as the raw material due to their easy availability and better film forming properties. Glycerol was chosen as the plasticizer because of their large plasticizing property for smaller quantities. Trial works were done based on manual composition. The film was prepared by mixing ingredients with water at 65-70℃ for 15-20 minutes. The mixture was then poured into petri plates and allowed to dry at room temperature overnight. Based on the observations of trail work, new composition formulated that starch varied from 0-2%, agar from 1-3% and glycerol from 0.5-1.5%. By using Box-Behnken Design (BBD) using Design Expert 7.0.0 Stat-Ease software, 17 runs were obtained. The Tensile strength, water vapour permeability, solubility and biodegradability were analysed for all 17 samples. An analysis of variance (ANOVA) was calculated for the model and all the responses in order to find its significance. The 3D graph was plotted for all responses using BBD. The optimization was done by setting goal for all independent and dependent variables. The optimized sample was obtained to be starch (1.39%), agar (2.91%) and glycerol (0.5%). For this composition, the tensile strength was 1165 N/m, modulus of elasticity was 314016.23 N/m², water vapour permeability was 61.25 g/m².day, solubility was 34.65%, biodegradability was 46.53% with desirability value as 0.861. The zone of inhibition for 4%, 8% and 10% garlic extract was measured against E. coli. It was found that 4% garlic extract was not effective. Films were prepared with 8%, 10% and 12%. The films prepared with 10% and 12% were found to have zone of inhibition of 1.3cm and 1.8cm respectively.

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