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Development of Sustainable Active Biocomposite Films from Rice Flour, Carrageenan, and Date Pit Extract for Enhanced Food Packaging and Shelf-Life Extension of Perishable Dairy Products

Shahad R. Mutlaq[1]*, Sabraa Saad Yaseen[2], Sara S. Faiad[3]

1,2,3-Department of Food Science, College of Agriculture, Tikrit University, Iraq

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*Corresponding Author E-Mail:
shahad.r.mutlaq@tu.edu.iq

ABSTRACT

The presence of conventional petrochemical based plastic packaging is presenting huge environmental issue and there is an urgent need to find alternatives or improvements to the current plastics, which are sustainable and can ensure the quality and safety of food products. The aim of this study is to develop new biocomposite active films (BPE) based on rice flour which is an agricultural by product and carrageenan, and reinforced by Date pit Extract (DPE) at different concentrations (0%, 1%, 2% and 3% w/w), with the use of glycerol as a plasticizer, by using the solvent casting technique. In the case of the best formulation (T2, 2% DPE), the mechanical performance was increased by around 44% reaching 25.8MPa (mechanical performance) and the lowest value of water vapor permeability (6.47×10^{-11} g/m·s·Pa). FTIR and SEM results showed that the hydrogen bonding was strong and the micro structures were uniform when 2% DPE was used; when 3% DPE was used, the molecular aggregation and phase separation were observed. In both series of active films, total phenolic content and radical scavenging activity (DPPH and ABTS) increased significantly and the films in both remained extremely biochemical stable after 12 weeks of storage. The soil burial test corroborated fast biodegradation (95 100% after 30 days) thus ensuring environmental sustainability. The preliminary application of T2 and T3 films on soft cheese showed that both films recorded TVC lower than the value of spoilage (7 log CFU/g) during the storage at 4 °C for 21 days.The application of T2 and T3 films onto soft cheese showed that, both films presented TTC values below the value of the spoilage (7 log CFU/g) during storage for 21 days in a fridge (4 °C), which extended the shelf life of the cheese by 2 4 days compared to T0 film. These films bring several advantages under industrial view point such as: (i) use of agricultural by products as renewable packaging materials, (ii) delayed release of bioactive phenolic compounds and consequently improvement in perishable dairy products shelf life, (iii) minimizing food waste and economic loss, and (iv) aligning packaging with the increasing consumer interest in clean label and eco-friendly packaging materials. This finding confirms the developed DPE loaded rice flour/carrageenan films as good material for sustainable and active food packaging, a material that could be economically used to decrease the plastic waste and increase the shelf life of perishable foods and intercepts the consumer demand for environmental friendly packaging.

1- INTRODUCTION

Conventional methods of food packaging are mainly non biodegradable petrochemical polymers, resulting in millions of tonnes of plastic waste being thrown away each year, and significant environmental pollution resulting in this manner [1]. This has encouraged increasing interest on the use of sustainable and biodegradable materials that can jeopardize health and quality of food but with ecological number. [2, 3] Rice flour, a low cost agricultural byproduct, is a viable film material, but it has high WVP due to protein and lipid residues compared to film material of pure starch matrices [4]. To investigate the drawbacks, carrageenan, a sulfated polysaccharide from red seaweed, has been supplemented in starch matrices, which formed cohesive networks that increase tensile strength and decrease water vapor permeability [5, 6].

The use of natural phenolic compounds as antioxidants and antimicrobial agents, thermal stabilizers and non volatile, in these films, aiming to improve their functional properties, has increased in the last years [7, 8]. One by product of the date that is rich in antioxidant and antimicrobial phenolic compounds is date pit extract (Phoenix dactylifera L.), which makes up 10 15 wt % of the date weight [9]. The incorporation of these extracts into polymer matrices is shown to increase moisture resistance, water vapor permeability, on the other hand, water solubility reduces, but also increases the structural stability [9]. The effect of date pit extract as structural and physical reinforcer for co material of rice flour/carrageenan is still not investigated.

In the food industry, active biodegradable packaging offers more advantages: (i) the avoidance of the use of synthetic plastics, (ii) the ability to extend the shelf life of perishable foods by gradually releasing antimicrobial and antioxidant compounds, (iii) the reduction of food waste and of the associated economic losses, and (iv) the ability to respond to consumer requests for the production of clean label and environmentally-friendly products [10, 11]. Furthermore, this approach offers good potential for a circular economy and the use of by-products of the agriculture industry (rice flour, date pits) as raw materials makes the entire process economically viable.

Hence, the current research was designed to formulate novel composite bio-Films from "Rice flour (RF)" and "Carrageenan" (CGN) with enhanced level of Date Pit (DP) extract and to investigate the effect of this combination on the physical, mechanical, structural, antioxidant and biodegradation properties of produced films in order to be used for packaging perishable/main mass food especially soft cheese.

2- MATERIALS AND METHODS

2.1. Raw Materials

The broken rice (*Oryza sativa* L.) was collected from a local rice mill, washed, dried at 50 °C, milled by roller mill and sieved with 100-mesh size (150 µm). Carrageenan (κ type) and glycerol (plasticizer) were obtained from the commercial sources. Pits of dates were harvested from the date processing facilities in the area.

2.2. Preparation of Date Pit Extract (DPE)

The date pits were cleaned, washed and dried at 50 °C then ground to a fine powder (<500 µm) and extracted with aqueous ethanol (70%, v/v) at a ratio of 1:10 (w/v), by continuous stirring at 40 50 °C for 60 120 minutes [9]. The extract was cleaned in whatman No. 1 filter, concentrated with a rotary evaporator at <40 °C and kept at 4 °C in light proof container.

2.3. Characterization of DPE

Folin Ciocalteu Method [12] was used to measure total phenolics. The antioxidant activity was measured by DPPH and ABTS radical scavenging assays [13].

2.4. Conducting of the film preparative experiments.Film preparative experiments were carried out.

The solvent casting method was used. Rice flour was gelatinized in rice flour suspension (3 4% w/v) for 15 20 minutes at 80 90 °C. The dispersion time of carrageenan (1 2% w/v) in water at 70 80 °C was tested. The suspensions were made with a mixture of glycerol (25 30% w/w) and the rest of the suspension was made up of water. DPE was incorporated at 0% (T0, control), 1% (T1), 2% (T2), and 3% (T3) of total biopolymer. Solutions were vacuum degassed, cast onto acrylic plates and dried at 35 40 °C for 18 24 h followed by conditioning at 25 °C, 50 ± 5% RH for 48 h.

2.5. Film Characterization

Physical properties: Thickness (digital micrometer), moisture content (gravimetric), water solubility, water vapor permeability (ASTM E96 cup), color (colorimeter), and opacity (UV Vis at 600 nm).

Mechanical properties: Tensile strength (TS), elongation at break (%E), and elastic modulus (ASTM D882) were determined by a texture analyzer.

Chemical properties include Total phenolic content (Folin Ciocalteu), antioxidant activity (DPPH, ABTS), FTIR spectroscopy and SEM for microstructure.

Biodegradability: Burial in soil put to test (natural garden soil, 25 °C, 90 ± 2% RH) for 30 days with weight loss tracked.

2.6. Apply to soft cheese such as cheeses made with milk.

Following the films were continuously developed and wrapped with fresh soft cheese samples and kept at 4 °C for 21 days. To evaluate the ability of the films to control microbial spoilage, total viable bacterial counts were measured on the films at 0, 7, 14 and 21 days.

2.7. Statistical Analysis

Triplicate experiments were done for all experiments. The data were reported as one way ANOVA, Duncan's multiple range test ($P< 0.05$). A summary expression ANOVA table (Table 1) is supplied to demonstrate the summary details of effect main on film properties in the function of DPE focus.

3-RESULTS AND DISCUSSION

3.1. The physical properties of the films.

The data on physical properties are summarized in Table 2. The thickness of film prepared with DPE was also significantly ($P < 0.05$) increased with incorporation into the film, as was the moisture content and water solubility content of the film. The water vapor permeability (WVP) of the materials reduced from 9.15×10^{-11} g/m·s·Pa at T0 to a minimum of 6.47×10^{-11}g/m·s·Pa at T2 (29% reduced) and then increased again at T3. The decrease of the WVP for the rice flour/carrageenan matrix at 2% DPE is attributed to the hydrogen bonding between the phenolic hydroxyl of DPE and the hydroxyl and sulphate group of the rice flour/carrageenan matrix which lead to the formation of a more compact and cohesive polymer network. This is an important property for food packaging with the lower value of WVP, less moisture will be transferred from the food to the environment; thus, food product quality and shelf life will be kept [4, 5, 9].

Table 1. Summary ANOVA Table for Main Effects ($P < 0.05$)

Property	df (Treatment)	df (Error)	F-value	p-value	Duncan Grouping
Thickness	3	8	12.45	<0.001	a, b, c, d
Moisture content	3	8	18.67	<0.001	a, b, c
Water solubility	3	8	15.23	<0.001	a, b, c
WVP	3	8	22.89	<0.001	a, b, c
Tensile strength	3	8	28.34	<0.001	a, b, c, d
Elongation at break	3	8	14.56	<0.001	a, b, c
Elastic modulus	3	8	19.78	<0.001	a, b, c
Total phenolic content	3	8	345.67	<0.001	a, b, c, d
DPPH scavenging	3	8	287.45	<0.001	a, b, c, d
ABTS scavenging	3	8	298.12	<0.001	a, b, c, d

Table 2. Physical characteristics of rice flour/carrageenan films (mean ± SD, n=3)

Treatment	DPE (%)	Thickness (mm)	Moisture content (%)	Water solubility (%)	WVP ($\times10^{-11}$ $g\cdot m^{-1}\cdot s^{-1}\cdot Pa^{-1}$)	ΔE*	Opacity (A600/mm)
T0 (control)	0	0.081 ± 0.004[a]	15.8 ± 0.7[a]	33.4 ± 1.6[a]	9.15 ± 0.42[a]	—	1.86 ± 0.09[a]
T1	1	0.086 ± 0.003[ab]	13.9 ± 0.6[b]	29.2 ± 1.3[b]	7.84 ± 0.38[b]	3.42 ± 0.21[a]	2.65 ± 0.14[b]
T2	2	0.091 ± 0.004[bc]	12.1 ± 0.5[c]	25.6 ± 1.1[c]	6.47 ± 0.33[c]	6.05 ± 0.29[b]	3.78 ± 0.19[c]
T3	3	0.097 ± 0.005[c]	13.4 ± 0.6[bc]	28.9 ± 1.4[b]	7.12 ± 0.36[bc]	9.87 ± 0.44[c]	5.46 ± 0.24[d]

Values with different superscript letters within the same column differ significantly (Duncan's test, P < 0.05).

3.2. Mechanical Properties

The tensile strength (TS) changed significantly ($P < 0.05$) from 17.9 MPa (T0) to 25.8 MPa (T2) which is approximately 44% increase, followed by a decrease to 22.4 MPa (T3). Enlongation at break also showed an increase from 39.4% (T0) to 47.6% (T2) and then dropped to 36.2% (T3). As seen the Elastic Modulus was high first at T0 (198 MPa), then at T2 (276 MPa), which showed greater 'stiffness', and decreased at T3. The better improvement (2%) DPE is said to be due to the good intermolecular hydrogen bonding between the phenolic components of the extract and polysaccharide chains existing in the network, which allows for a more complete partitioning of stresses [5, 9]. While DPE is declining, it is indicated at 3%, as a result of molecular aggregation and phase separation, the formation of stress concentration surfaces and parts that catalise crack formation which has been confirmed by SEM images showing aggregates and discontinuities within the T3 films. All things considered, from a food packaging viewpoint, the strength and flexibility at 2% DPE is perfect—to be able to handle and transport the pack without breaking, and then have enough flexibility so it doesn't break while storing and using the packaging.

Table 3. Mechanical properties of the films (mean ± SD, n = 3)

Treatment	DPE (%)	Tensile strength (MPa)	Elongation at break (%)	Elastic modulus (MPa)
T0 (control)	0	17.9 ± 0.8[a]	39.4 ± 1.9[ab]	198 ± 11[a]
T1	1	21.3 ± 0.9[b]	43.8 ± 2.1[bc]	232 ± 13[b]
T2	2	25.8 ± 1.1[c]	47.6 ± 2.4[c]	276 ± 15[c]
T3	3	22.4 ± 1.0[b]	36.2 ± 1.8[a]	244 ± 14[b]

Values with different superscript letters within the same column differ significantly (Duncan's test, P < 0.05).

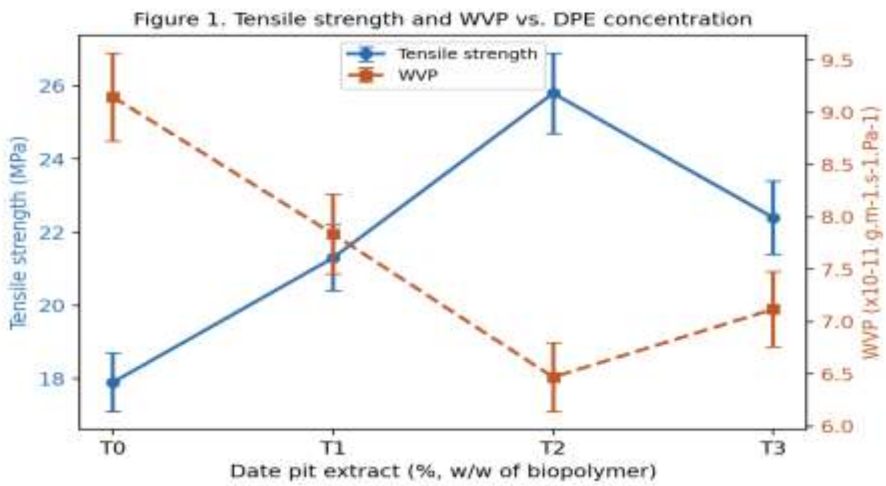

Figure 1. Mechanical properties of films (T0–T3): Tensile strength and WVP

(To be included: Bar chart comparing tensile strength (MPa) and WVP ($\times 10^{-11}$ g/m·s·Pa) across treatments T0–T3. Error bars represent standard deviation. Different letters indicate significant differences at P < 0.05.)

3.3. Chemical and Antioxidant Properties

As presented in table 4, both the total phenolic content and antioxidant activity increased with the dose of the DPE incorporated which is dose dependent. The DPPH scavenging activity was found to be increasing from 8.3% at T0 to 84.1% at T3 while the ABTS scavenging activity was observed to be increasing from 6.7% at T0 to 79.3% at T3. After 12 weeks' accelerated storage at 25 °C, activity of T3 was 55.8% (about 66% retention) which shows good stability and can be used for long term food preservation [9]. Hydrogen bonding interactions between DPE phenolic hydroxyls to polysaccharide matrix as shown by broad and shift in O H stretching region from 3287 cm^{-1} (T0) is confirmed by FTIR at T3. This was confirmed by the SEM micrographs, which revealed smooth and homogeneous surfaces for T0 and T2, while T3 displayed the presence of aggregates and phase separated regions at higher DPE loadings, as evidenced by the SEM micrographs.

Table 4. Total phenolic content and antioxidant activity of the films (mean ± SD, n = 3)

Treatment	DPE (%)	Total phenolic content (mg GAE/g film)	DPPH scavenging (%)	ABTS scavenging (%)
T0 (control)	0	0.42 ± 0.05^{a}	8.3 ± 1.1^{a}	6.7 ± 0.9^{a}
T1	1	5.16 ± 0.34^{b}	46.2 ± 2.8^{b}	41.5 ± 2.4^{b}
T2	2	9.83 ± 0.51^{c}	69.7 ± 3.2^{c}	64.8 ± 3.0^{c}
T3	3	14.27 ± 0.68^{d}	84.1 ± 3.6^{d}	79.3 ± 3.4^{d}

Values with different superscript letters within the same column differ significantly (Duncan's test, P < 0.05).

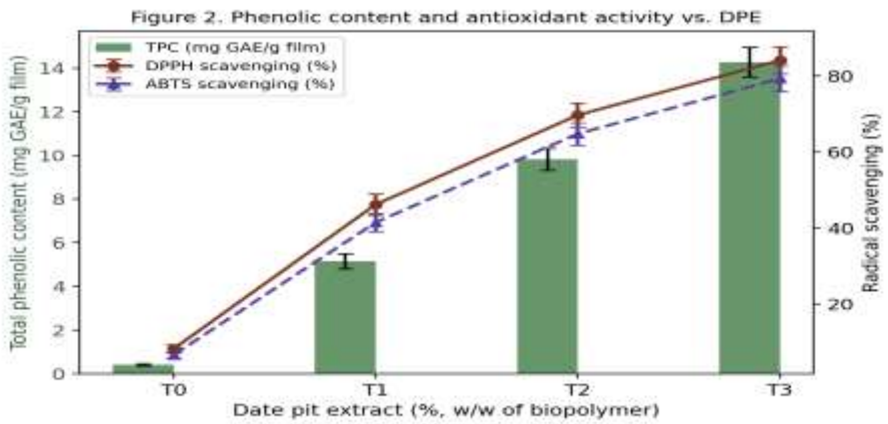

Figure 2. FTIR spectra of DPE-loaded treatments (T0–T3)

(To be included: Overlay of FTIR spectra showing the progressive broadening and shift of the O-H stretching band from 3287 cm⁻¹ (T0) to approximately 3255 cm⁻¹ at T3, indicating hydrogen bonding interactions.)

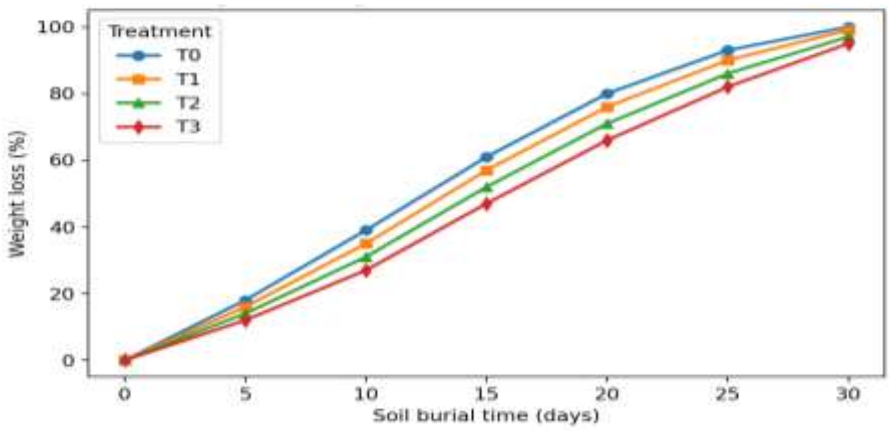

Figure 3. SEM micrographs of film surfaces (T0–T3)

(To be included: SEM images showing smooth, homogeneous surfaces for T0–T2, and visible aggregates and phase-separated regions for T3.)

3.4. Biodegradability

The weight loss due to soil burial is presented in Figure 4. Rapid biodegradation of all treatments was found, with weight loss of more than 47% on day 15. All films were 95 100% weight loss by day 30. The control (T0) degraded most rapidly (100% degradation after 30 days) and a slight effect related to the level of DPE was noted, which may be attributed to the moderate, but significant, antimicrobial effect of phenolic compounds. Their rapid biodegradability has been established, thus proving that the films' use is environmentally friendly and provides an alternative for using non-degradable plastics for food packaging.

Figure 4. Soil burial for 30 days, weight loss (%) of the films.

To be included: Line graph - weight loss (%) as a function of days (0-30) for T0-T3. Standard deviation (Error bars).

3.5. Extensions on this product are under development.This product is being developed for extension on soft cheeses.

The new film products were proven to be of interest for use in the food industry in a preliminary run with soft cheese. Total viable bacteria counts of films with 2% DPE (T2) and 3% DPE (T3) remained below the spoilage level (7 log CFU/g) during the 21 day period of the refrigerated storage. A control film (T0) did not reach this point until after 14 days. The extended shelf life of 2 4 days is explained based on the reduction of the microbial growth ability due to the presence of phenolic compounds released slowly from the films, disrupting the microbial membranes and inhibiting the enzymes involved in the microorganism growth [9, 14]. It is an economically important extension particularly to the dairy industry in light of the product losses that can be avoided and distribution time extended, along with the demand for minimally processed, naturally preserved foods. It is even more cost effective and sustainable solution, since the agricultural by products (date pits, rice flour) can be used as raw material.

4- DISCUSSION

The results indicate that date pit extract (DPE) can be used as an effective reinforcing and active agent in rice flour/carrageenan biocomposite films. The concentration of DPE is optimized at 2% (T2), which offers a suitable combination of mechanical strength (increase in tensile strength by 44%), barrier property (a decrease in water vapour permeability of 29%) and antioxidant activity, making it suitable for use in food packaging applications. It has been considered the hydrogen bonding among polysaccharide matrix with the phenolic hydroxyl groups of DPE, which leads to the generation of compact and cohesive hydrogen bond network [5, 9]. Several properties of 3% DPE have decreased with the increase in molecular aggregation and phase separation, suggesting an optimum quantity and beyond which this reinforcing effect is lost.

A positive angle for phim From a food industry standpoint, these films have a number of benefits:

1. They are sustainable, using renewable by-products of the agricultural industry (such as rice flour and date pits) which helps minimize waste and the use of fossil fuels.
2. Active functionality: Antioxidant and antimicrobial effects of DPE can be used for providing an extended shelf life for perishable foods such as cheese, meat, and fresh products, which can inhibit their oxidation process and microbial growth.
3. Biodegradability – quickly breaks down in soil, so these films do not cause long term pollution.
4. Economic viability: The technology is easily accessible to the food manufacturers as it reduces food waste and food distribution is extended in use of cheap raw materials.

The findings are similar to the previous studies related to polyphenol fortified polysaccharide films [9, 10, 14]. The current research contributes to the body of knowledge by showcasing the application of rice flour, carrageenan and DPE in one biocomposite and its synergistic behavior effect.

5- CONCLUSION

A novel biodegradable active film made from rice flour, carrageenan and date pit extract was successfully prepared in this study. The best mechanical properties (25.8 MPa, 44% increase) along with the least water vapour permeability (6.47×10^{-11} g/m·s·Pa, 29% decrease), were provided by the optimum formulation (2% DPE and T2). The high antioxidant activity (69.7% DPPH scavenging), as well as the rapid biodegradability (95 100% weight loss in 30 days) were also attained. Preliminary application on soft cheese showed that T2 and T3 films were effective in keeping the microbial counts below the spoilage level for 21 days resulting in shelf lives 2 4 days longer than the control films. These results have confirmed that the produced biocomposite films are a good potential for sustainable and active packaging systems, which is less economical and environmentally friendly than coated-packaging plastics. In order to make full use of the commercial potential of this packaging technology, further work is recommended, such as large scale production, migration analysis and application to other food products (meat, fresh produce).

The developed films could be of value from an industrial point of view to address three linked challenges: (i) the reduction of plastic waste by using renewable agricultural by-products; (ii) the shelf-life of perishable dairy products by obtaining a controlled release of bioactive phenolic compounds; (iii) consumer demand for packaging with a "clean" label and that can be considered environmentally friendly. The marketability of this technology has been proved with the use of the low cost raw materials (rice flour, date pits) as well as the shelf life extension (2 4 days).

Conflict of Interest

The authors declare that they have no conflict of interest.

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This research received no external funding.

Data Availability

All data supporting the findings of this study are available within the manuscript.

Ethical Approval

This study did not involve human participants or animals. All experimental procedures were conducted in accordance with institutional and laboratory biosafety guidelines.

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