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Additive properties of mint weed in polyfilms

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ABSTRACT

Biopolymer films have been regarded as potential replacements for synthetic petrochemical based polymers for different uses mainly in pharmaceuticals and packaging applications in view of strong awareness towards more environmental friendly materials. Polysaccharide-mucilage and lignin concentrate were isolated from the mint weed (*Hyptis suaveolens*). Potato starch was modified and starch-lignin-mucilage-polyvinyl alcohol was prepared in different composition from lignin and mucilage isolates of *H. suaveolens*. The physical properties like film appearance, opacity, swelling per cent and mechanical properties namely tensile strength and elongation per cent of polyfilms were measured. Acetylated starch with lignin isolates showed good elongation % (17.43±0.24) in comparison to native starch films (2.62±0.11). Acetylation increased the tensile strength while *Hyptis* lignin increased the elongation per cent of films. The tensile strength of mucilage and polyvinyl film blend was found to be decreased significantly. This study revealed that lignin and mucilage blends are compatible with starch and synthetic polymers which could be advantageous for cost reduction with improved properties and enhanced increase range of application.

Key words: Additive, Biopolymer films, Elongation per cent, *Hyptis suavelones*, Mucilage, Lignin

Starch has received considerable attention because of its complete biodegradable nature and low cost (Arvanitoyannis *et al*. 1994, Liu 2005, Garcýa *et al*. 2006). Starch film properties can be influenced by the addition of different additives as fillers or modified chemical reactions and can also be readily converted chemically and biologically into many useful and diverse products such as paper, textiles, adhesive, beverages, confectionaries, pharmaceuticals and plastics (Agbo *et al.* 2010). Arvanitoyannis *et al.* (1998) and Lafargue *et al*. (2007) studied formulation of composite films by mixing with other biopolymers, such as gelatin.

Hyptis suaveolens Poit, the pig nut or mint weed, a member of Lamiaceae family and native of tropical regions of Mexico, West Indies and South America, has become naturalized in tropical parts of Africa, Asia and Australia. It has also well established in India and is considered as a potent alien invader for reducing biodiversity and palatable grasses. However, *H. suaveolens* also has good medicinal value owing to the presence of essential oil, a characteristic feature to the family Lamiaceae. But, due to these essential oils, it is unpalatable to animals. It is known to be used in several traditional medicines for the treatment of various illnesses and has been found to possess significant pharmacological applications (Kuhnt *et al*. 1995). It is also a potential source of lingo-cellulosic material and mucilage which can be utilize as an additive in polymeric materials to change the properties according to their applications. Hitherto, this properties was unexplored, therefore, in the present study, we have studied the compatibility of *Hyptis* lingocellulosic and mucilage material with starch and polyvinyl alcohol for use in making polyfilms.

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MATERIALS AND METHODS

The study was carried out during 2011 and 2012 at Non Wood Forest Produce Lab of Tropical Forest Research Insitute, Jabalpur located at 23.1667° N, 79.9333° E. The aerial parts of the weed was collected from the forest and roadside. The material was dried in shade

Isolation of starch: Starch was isolated using a procedure of Kim *et al*. (1995). The potato tubers were peeled, cut into 5-6 cm cubes and immediately rinsed in sodium sulphate solution and then macerated at low speed in a Waring blender for two minutes. The homogenate was consecutively sieved. The white-starch sediment was washed several times with water and then centrifuged. The white-starch sediment was dried in an oven at 40^0 C for 48 h.

Modification of starch: Acetylated and hydroxypropylated products of starch were prepared by the modification according to procedure reported by Jarowenko (1986).

Extraction of lingo-cellulosic material: Aerial part of *Hyptis* was collected and leaves were removed. Dried twigs was used as the source of lignin-cellulosic material. Fifty gram material was digested in 500 ml of 1% NaOH solution for 5 hours at around 100 ^{0}C. The solution was filtered and filtrate was concentrated to 75 ml. The weight of dry extract was found to be 9 g. *i.e,* 75 ml solution had almost 3 gm of lignin.

Extraction of mucilage: Fruits or seeds of H. suaveolens were used for isolation of mucilage. Fruits were soaked in warm water for 4 h, boiled for 2 h and kept aside for 2 h for release of mucilage into water. The material was squeezed in a muslin cloth and centrifuged. Equal volume of ethyl alcohol was added to filtrate to precipitate the mucilage. The separated mucilage was dried in oven at about 45 ^{o}C and powdered. The powdered mucilage was stored in desecrator until further use (Malviya *et al.* 2010).

Preparation of starch-lignin-mucilage polymer: Starch-based films were prepared by casting technique. Polyol, glycerol was used to plastisize the films. Films were prepared using suspension of plasticizer, potato starch, distilled water, lignin and mucilage isolate. Four gram of starch was mixed with 75 ml water with constant stirring at 100 ^{0}C until the mixture turned semi-viscous. Ten ml of lignin solution was added to the semi-viscous starch solution. The starch-additive mixture was stirred vigorously for another ten minutes. Later, the polymer mixture was spread on a plastic coated plate and left to cure. Film forming suspension was heated with continuous mixing by a magnetic stirrer and at short intervals by hand with a glass rod to above 90 ^{0}C- 100 ^{0}C to obtain film forming solution. The solution was kept at 90 ^{0}C for 5 minutes before letting it cool down to 50 ^{0}C. Air bubbles formed during heating were removed from solution before casting on glass plate. Films were removed from plate after curing in an oven at 35 ^{0}C. Similarly, films of polyvinyl alcohal, modified starch and mucilage were prepared.

Film solubility in water, oil and hexane: The film solubility was determined by following procedure described by Gontard *et al.* (1992). The film pieces of 2 x 2 cm were cut from each sample and stored in a dessicator with silica gel for 7 days. Samples were weighed and placed into test beakers with 80 ml deionized water and were maintained under constant agitation at 200 rpm for 1 h at room temperature (approximately 25 ^{0}C). After soaking, the remained pieces of film were collected by filtration and dried again in an oven at 60 ^{0}C to constant weight. The percentage of total soluble matter (% solubility) was calculated as follows:

% Solubility= (Final weight- initial weight of film/ Final dry weight)x100

Film opacity: Film opacity was determined using a procedure described by Gontard *et al.* (1992). The film sample was cut into a rectangle and placed on the internal side of a spectrophotometer cell. The absorbance spectrum (400–700 nm) was recorded for each sample using spectrophotometer. Film opacity was defined as the area under the recorded curve determined by an integration procedure. The opacity was expressed as absorbance units per nanometers (AU nm).

Film appearance: Homogeneity and appearance of the films were examined by visual observation and optical microscopy.

Thickness of Films: Thickness of the films was measured with the help of micrometer.

Mechanical Testing: Mechanical properties *i.e.* tensile strength, the percentage elongation at break and young's modulus were evaluated. The 12 x 2 cm^2 sized polymeric films were cut and subjected for the mechanical analysis. The tensile strength, tensile modulus, and elongation were measured using an Instron Universal Testing machine by adopting IS 2508 method at Central Institute of Plastic Engineering, Bhopal (M.P.).

RESULTS AND DISCUSSION

The transparent, translucent, homogeneous, thin and flexible films were obtained with different blends from native and chemically modified starches.

The oven-dried lignin blended films had a pale yellow colour. No pores or cracks were detected in films prepared by filmogenic suspension containing lignin and mucilage as an additive. The thickness of the tested films varied 0.14-0.16 mm.

The film opacity and swelling in different medium are the critical properties of a film. Opacity of film in different blends and swelling in different solvents are given (Table 1). Significant variation was observed in swelling % of different blends in water and oil, used as solvent. All films were swelled in water except acetylated starch and polyvinyl blend. The swelling properties of films in oil was decreased with the addition of *Hyptis* mucilage and lignin. Transparent films were characterized by low values of the area below the absorption curve.

Table 1. Physical properties of native and chemically modified starch films with and without additives

Film composition	Swelling %			Film opacity [AU nm]
	Water	Haxane	Oil	
S+P	0.15±0,02	0.05%	0.33±0.01	100±0.00
Ac.S + P	0.05±0.01	Resistive	0.250.03±	100±0.00
Ac.S + H + P	0.66±0.02	Resistive	0.09±0.00	0.00±0.00
AcS + PVOH	0.00±0.00	Resistive	0.03±0.04	100±0.00
HPS+ H+ P	1.24±0.07	Resistive	0.12±0.03	0.00±0.00
S+mucilage + P	0.24±0.03	Resistive	0.17±0.01	0.00±0.00
PVOH + mucilage + P	1.07±0.08	Resistive	0.14±0.02	64.540.01
LSD(P=0.05)	0.35	NS	0.07	-

S-starch,Ac- acetylated, PVOH-Polyvinyl alcohol, P-plasticizer, HP-Hydroxypropylated, H-*Hyptis*

The tensile strength and elongation at break of native and modified starches and polyvinyl blended films are shown in Table 2. Results indicated that the addition of different additives caused differences in both tensile strength and elongation of polysaccharide films as well as polyvinyl alcohol.

The maximum tensile strength (427.5 kgf/cm^2) was observed in modified followed by PVOH blend (391.79 kgf/cm^2) and 10.42 mm and 1.85 mm elongation, respectively. The maximum elongation (17.43 mm) was observed in film blends with *Hyptis* lignin isolates corresponds to 159.41 kgf/cm^2 tensile strength but minimum tensile strength (92.67 kgf/cm^2). Significant difference was observed in different blends with hyptis lignin isolate and mucilage. Mucilage reduces tensile strength of polyvinyl alcohol in comparison to acetylated starch and polyvinyl films.

Baumberger *et al*. (1998), studied starch-lignin films, also found that lignin improves water resistance. Without additional plasticization, lignin increases the brittleness of both films and foams. The present results can be compared with those of Baumberger *et al*. (1998) and Stevens *et al*. (2007) prepared starch-kraft lignin pre-extruded molded films containing only water as a plasticizer found that introduction of lignin increase the hydrophobicity of starch-lignin films and reported that hydrophobic nature of films can reduce by the addition of plasticizer. They observed tensile elongations at break of 1.0-1.5%.

Table 2. Mechanical properties of films of different composition

Polymeric blends	Width (mm)	Elongation at break(mm)	Tensile strength (kgf/cm^2)
S+P	25.1	2.62±0.11	99.7±8.3
Ac.S +P	25.5	1.85±0.09	427.5±11.2
AcS+H+P	25.1	17.43±0.24	159.4±16.7
Ac.S +PVOH+ P	25.6	10.42±0.90	391.8±12.2
HPCA+ H+ P	25.5	2.73±0.23	310.3±10.1
S+mucilage+ P	25.2	14.24±1.2	105.4±8.1
PVOH + mucilage + P	25.5	6.89±0.39	57.2±9.2
LSD (P=0.05)		2.31	28.9

S-starch,Ac- acetylated, PVOH-Polyvinyl alcohol, P-plasticizer, HP-Hydroxypropylated, H-*Hyptis*

H. suaveolens is a good source of polysaccharide-mucilage. Its lignin and mucilage showed compatibility in polymeric blends.

The possible advantage of using other polymers with native starch granules lies in that it can be deformed and distributed in the blending process and enhance other ways of utilization.

Starch-based film incorporated with lignin and mucilage were compact, translucent and presented good flexibility and elastic than native starch-based films In addition they were easy to handle when dried off.

The films containing lignin possessed good mechanical. However, it affect on opacity of the films. Starch is a widely used biopolymer, but lignin-starch systems are a relatively unexplored area in biopolymer research.

The results indicate that blending of starches and polyvinyl alcohol with other polymers could be advantageous for cost reduction with improved properties when compared to native starches of species. Thus the chemical structure of the polymer was changed by blending it with other polymers to improve its physical characteristics thus widening range of application. Polymer blending offers interesting possibilities of preparing inexpensive biodegradable materials with useful mechanical properties.

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