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Investigation on Physical-Mechanical Properties of Natural Polymer Films

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ABSTRACT: Coating of the dosage forms particularly for pulsatile release plays important role for controlling the lag time prior to release of the drug. In this study, novel natural coating polymeric material was isolated from the seeds of higher plant *Delonix regia* family-Fabaceae. Isolated polymer was characterized by X-ray diffraction study (XRD), Differential scanning calorimetry (DSC) and Scanning electron microscopy (SEM). Films of this novel polymer were prepared and evaluated for various physical and mechanical properties by tensile tester and bursting strength apparatus. Force, breaking strength, bursting strength and elastic modulus were investigated with respect to the type of polymer and plasticizer used. The prepared films were found to be flexible, had a high strain and burst with only small cracks and was comparable with synthetic polymers which are frequently used for pulsatile as well as sustained release drug delivery system. The microbial flora in the colon degraded the gallactomannan present in the polymer and thus releases the drug in the colon. Therefore, this natural polymer may be a promising candidate for pulsatile drug delivery system which releases the drug at colonic site after a predetermined lag time.
Key Words: *Delonix regia*, Natural Polymer films, Physical-mechanical properties, Pulsatile release.

INTRODUCTION

Water-insoluble polymers are applied as coatings to solid dosage forms either from organic polymer solutions or preferably, because of the absence of organic solvents, from aqueous colloidal polymer dispersions. The permeability and mechanical properties of such coatings are often determined on free polymer films prepared by casting or spraying techniques [1]. PEG 400 was incorporated as plasticizer for reducing the brittleness of the films formed and provides them elasticity.

The major advantages of incorporation of a plasticizer in film coating systems include enhancement in film formation, reduction in film brittleness, better adhesion to the tablet surface, and reduction in logo bridging [2].

Earlier we have developed pulsatile drug delivery system based on insoluble capsule body filled with theophylline microspheres and sealed with swellable natural polymer plug isolated from the endosperm of seeds of higher plant *Delonix regia* family-Fabaceae. Formulated pulsatile drug delivery system was evaluated for in vitro drug release study, which showed release might be consistent with a release time expected to deliver drug to the colon depending on the thickness and hardness of the hydrogel plug [3].

In this study, attempt has been made to investigate physicochemical and mechanical properties of isolated polymeric material from the natural source as a function of various polymer film parameters. The drug release may takes place in colon due to degradation of gallactomannan present in polymer films by colonic micro flora at predetermined time period depending on the level of coating of polymer on tablet core.

MATERIALS AND METHODS

Materials

The polymers used in this study for comparing the mechanical properties of natural polymer were: hydroxypropyl methylcellulose (HPMC K4M) received as a gift sample from Colorcon Asia Pvt Ltd., Goa, India. Eudragit S 100 and Eudragit L 100 were supplied as a kind gift from EvonicDegussa India Limited, Mumbai, India. All other reagents were of analytical grade and used as received.

Methods

Isolation of natural polymer [3]

The seeds of the higher plant *Delonix regia* were collected and subjected to boiling for 5-6 h. Seed coat were removed and the mesosperm was separated which was then transferred into beaker containing distilled water and boiled again to get the viscous mass. This viscous polymeric mass was then allowed to dry in an oven till it dried completely. The dried polymeric material was then subjected to size reduction in multimill and sieved through sieve shaker using 60 # sieve to get the desired particle size.

Characterization of powdered polymer

Powdered natural polymer was characterized by photo microscopic study, Scanning electron microscopy (SEM), Differential scanning calorimetry (DSC) and X-Ray diffraction studies (XRD).

Photo Microscopic Study [3]

The powdered *Delonix regia* was placed on glass slide and a drop of cedar wood oil was added to disperse the *Delonix regia* uniformly which was then covered with cover slip to the slab surfaces with double-sided adhesive tapes and then a series of digital images of films were captured using a Motic DMWB2-223 digital microscope fitted with 1/3 CCD camera imaging accessory and using Motic Images 2000 (2.0 Version) image analysis software.

Scanning Electron Microscopy (SEM)

The SEM photographs of *Delonix regia* were obtained by scanning electron microscope (JSM 6390, JEOl, USA) with 10 kV accelerating voltage.

Differential Scanning Calorimetry (DSC)

DSC curve of natural polymer was obtained by a differential scanning calorimeter (DSC, Mettler Toledo, USA) at a heating rate of 10 °C/min from 30 to 350°C in nitrogen atmosphere.

X-Ray Diffraction Studies (XRD)

Powder XRD patterns of natural polymer was recorded using diffractograms (PW 1140, Mettler Toledo, USA) and Cu-kα radiation. Diffractograms were run at a scanning speed of 2°/mm and a chart speed of 2°/2 cm per 2 θ.

Preparation of Polymeric Films

Powdered polymer (5g) was weighed and transferred to beaker containing 100 ml of distilled water. This was then subjected to boiling till it becomes sufficiently viscous. This viscous solution (15 ml) was then poured uniformly onto previously lubricated petri plates (Internal diameter 9 cm) for preventing adherence of films to surface of the plate. These plates were then placed in an oven for 1 h at 50°C. After 1 h, films were removed with the help of stainless steel spatula and were evaluated for various properties.

Physical-mechanical Properties of Polymer Films

Film Thickness

The film thickness was measured at 3 different points with a micrometer screw gauge (Kayco, India).

Refractive Index

Refractive index of powdered polymer and all the three films with different concentration of plasticizer was determined by Abbe's refractometer at room temperature.

Moisture Absorption Capacity

Moisture absorption capacity of the polymeric film was determined by placing previously weighed films into the stability chamber at 75 % RH for 24 h. After 24 h, films were removed and excess of moisture on film surface was removed by slightly pressing the blotting paper over it. The films were then weighed. The difference in weight will give the amount of moisture absorbed by the film.

Swelling Index

Swelling index of the film was determined by reported method [4]. An area of 1 cm^2 (1x1 cm) of each film was dried in a oven at 50 ^{0}C for 24 h, dried film was accurately weighed and immersed in a flask containing 250 ml of different media at 37 ± 2^0 C. Swollen samples were withdrawn from the medium and weighed after removal of excess surface water by light blotting with Whatman's paper. First sampling was done at 15 min and subsequent sampling was done at time intervals of 30 min up to 165 min. The swelling index was then determined by following equation:

% S.I. = Ws-Wd / Wd ×100 (1)

Where Ws- Weight of film after swelling

Wd- Weight of dry film

Tensile Strength

Prepared films were subjected to tensile test using Ubique Tensile Tester (UTM, Universal Tensile Tester, Pune, India). Films were cut into strips of width 1cm and length 7 cm. These polymeric strips were then fixed between upper and lower platen of the tensile tester and the tensile strength was determined. Tensile strength, strain and % elongation was determined in the following manner [4]:

Tensile strength (σ) = Force or load (F)/ MA (2)

Where F is the maximum load and MA is the minimum cross-sectional area of the film specimen. Results were converted to megapascal units (MPa).

Tensile strain (ε) = $(L_0-L)/L_0 = \Delta L/L_0$,......(3)

% Elongation= ε ×100, … .(4)

L_0 refers to initial length of the film sample and L is the elongation at the moment of film break.

Bursting Strength

Bursting strength of the prepared film was determined using bursting strength apparatus (Bursting Strength

Tester, Ubique Systems, Pune, India) by fixing the film above the expanding rubber bulb. When the test starts this rubber bulb bulges which results into bursting of the film depending on its bursting strength.

Microscopic Study

The films were placed on glass slide and covered with cover slip to the slab surfaces with double-sided adhesive tapes and then a series of digital images of films were captured using a Motic DMWB2-223 digital microscope fitted with 1/3 CCD camera imaging accessory and using Motic Images 2000 (1.3 Version) image analysis software.

RESULTS AND DISCUSSION

Characterization of powdered natural polymer

Photo microscopic study

Photomicrograph of novel natural polymer shows lumps of irregular shape particles which indicate its amorphous nature which was further confirmed by scanning electron microscopy (Figure 1).

Scanning Electron Microscopy (SEM)

SEM photographs of powdered polymer, taken at different magnification, shows gum like mass which is devoid of crystalline structure (Figure 2).

Differential Scanning Calorimetry (DSC)

The DSC curve of *Delonix regia* polymer exhibited endothermic peak at 80°C. Exothermic peak at around 305°C shows degradation of the polymer (Figure 3).

X-Ray Diffraction Studies (XRD)

XRD pattern of the polymer has shown peaks with low intensity which confirms the amorphous nature of the polymer (Figure 4).

Physical-Mechanical properties of polymer films

Film Thickness

Thickness of the prepared polymeric film was measured at 3 different points using Micrometer Screw Gauge and the average of the reading has been taken as the thickness of particular films (Table 1).

Refractive Index

The refractive index of all the samples was found to be same (Table 1) indicating no interactions with plasticizer used.

Moisture absorption capacity

The results of the moisture absorption study reveals that the polymeric films of the novel natural polymer absorb sufficient amount of moisture (Table 1). The absorbed moisture may act as plasticizer and thus these films showed more tensile strength as compared to dried films.

Swelling Index

Swelling index of prepared films was determined at different time interval and in different solvents by the reported method [4]. Since the swelling index values remains approximately constant at 75 min and 105 min, last reading was taken after an interval of 1h (Table 2).

Tensile Strength

The result of the tensile strength determination indicates that the tensile strength of the *Delonix regia* film was found to be comparable to that of Eudragit S 100 & Eudragit L 100 film (EDG) (Figure 5). The coating film should possess a sufficient mechanical strength to avoid damage during manufacturing processes, storage, and transportation. Low elastic moduli were found to be advantageous to prevent the initiation and propagation of cracks and thus reduced the risk of dose dumping. The mechanical properties of coating film changes when they are immersed in release media. This is due to the uptake of water, which can act as a plasticizer of the polymer film, and also due to the leaching of additives in such films, including water soluble polymers and plasticizers.

Bursting Strength

The polymeric film of diameter 9 cm was fixed between the circular plates of bursting strength apparatus. The test was started and the bulb get bulged which results into bursting of the polymeric film with many cracks. The bursting strength was recorded. The film F2 when compared with film F1 showed significantly higher bursting strength as film F2 contains 5% PEG as a lubricant (Table 3).

Microscopic Study of Polymer films

Figure 2 shows the SEM micrograph of the Delonix regia, HPMC K4M and mixture of Eudragit S 100 and Eudragit L 100 film. The SEM photomicrographs of all the three films showed smooth surface of film (Figure 6).

CONCLUSION

Delonix regia biodegradable films were prepared successfully by spreading the known quantity of polymeric solution on prelubricated petridish. The mechanical properties of the prepared film were found to be comparable to those of other polymers like HPMC and Eudragit L 100 & Eudragit S 100 film prepared in the ratio of 1:2 w/w. From the results of the physicochemical and mechanical properties of the films it can be concluded that the natural novel polymeric material from *Delonix regia* may be natural and economical alternative for the formulation of pulsatile drug delivery system which releases drug rapidly in colon due to microbial degradation of gallactomannan present. However, extensive in vitro and in vivo study needs to perform to support the hypothesis.

Table 1. Physical Properties of *Delonix regia* Films without Lubricant (F1) and with Lubricant (F2)

Film code	Thickness (mm)	Refractive Index	Moisture absorption capacity (mg /inch^2)
F1	0.16 (0.01)	1.330	15.9 (1.4)
F2	0.16 (0.02)	1.330	19.1 (2.1)

n=3

Table 2. Swelling Index Study of *Delonix regia* film

Time (min)	Swelling index in different pH media		
	pH 1.2	pH 6.8	pH 7.4
0	28.2	22.1	42.2
15	116.3	185.1	91.9
45	371.6	506.3	407.1
75	375.2	551.6	423.7
105	364.5	556.0	420.7
165	360.0	556.1	420.0

Table 3. Mechanical Properties of Different Polymer Films

Film code	Bursting Strength (lb/inch^2)	Young's modulus	%E	Breaking Strength
F1	60 (2.2)	1.34	16.4	1.1 kg at 8.2 mm
F2	74 (3.1)	1.42	17.4	1.2 kg at 8.7 mm
EDG	39 (1.7)	18.57	2.8	1.8 kg at 3.3 mm
HPMC	54 (1.9)	5.45	6.6	2.6 kg at 1.4 mm

EDG= Eudragit S 100 : Eudragit L 100 in 1: 2 ratio

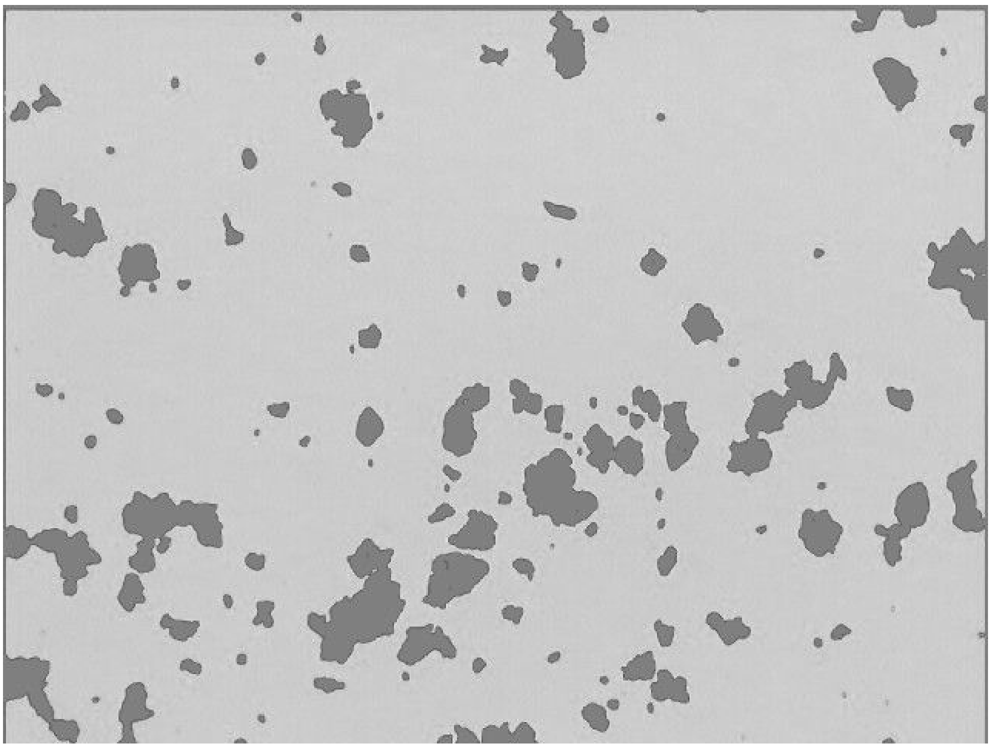

Figure 1. Photomicrograph of powdered *Delonix regia*

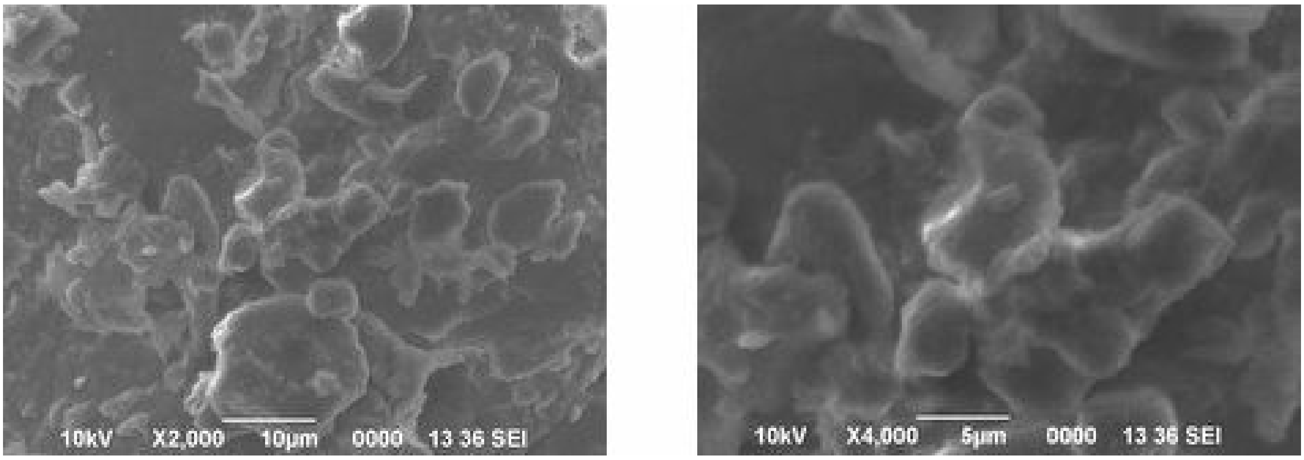

Figure 2. SEM Photographs of *Delonix regia* polymer at different magnifications

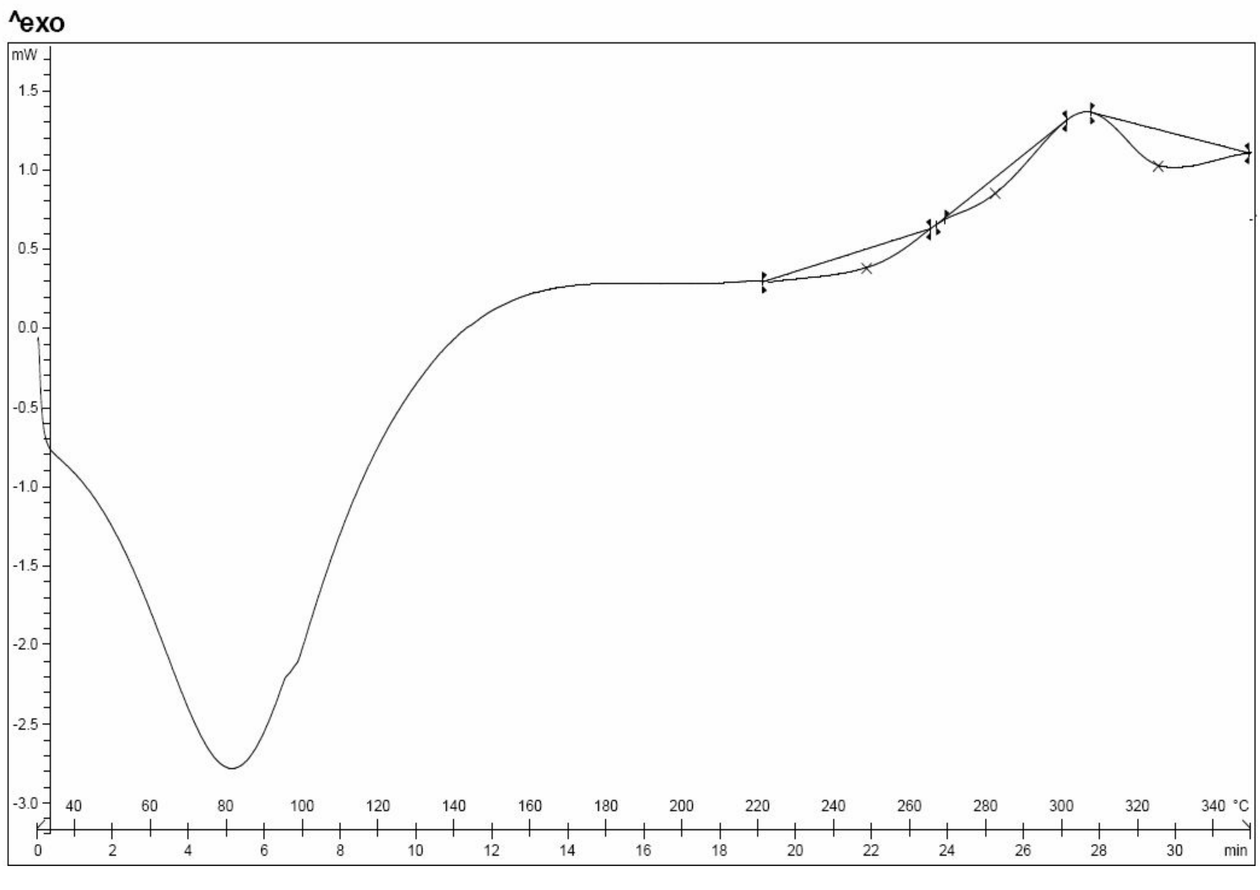

Figure 3. DSC profile of *Delonix regia* polymer

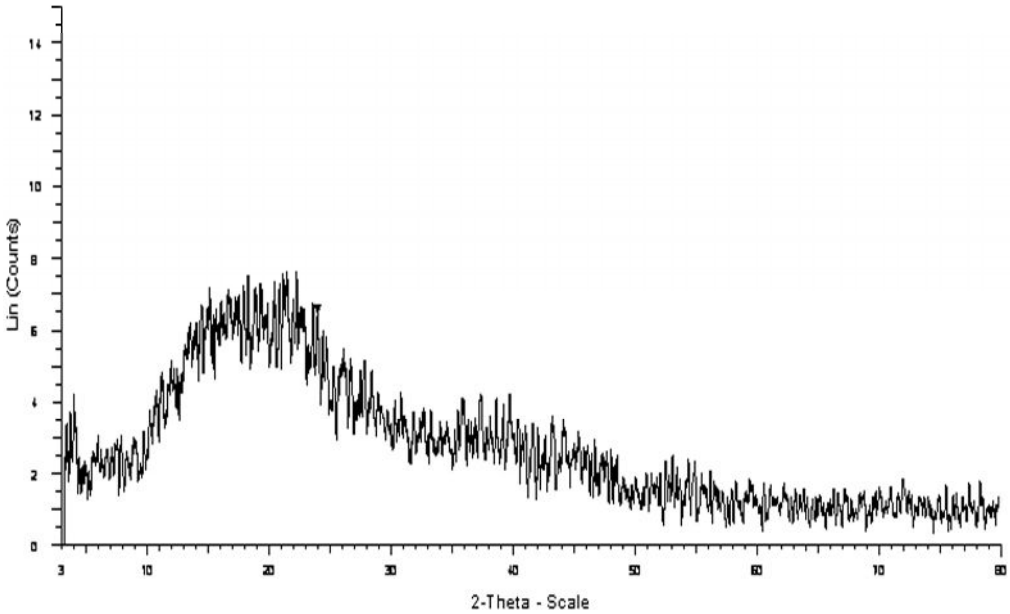

Figure 4. XRD pattern of *Delonix regia* polymer

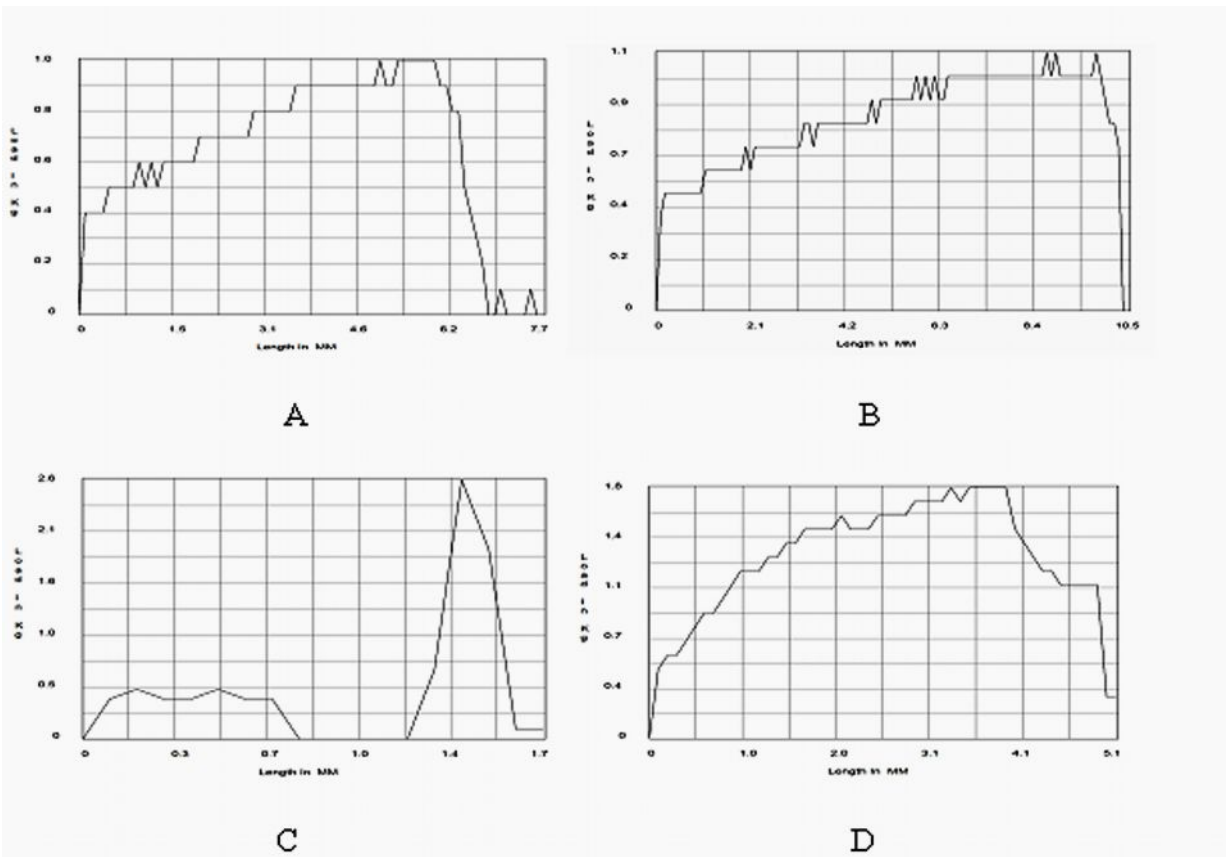

Figure 5. Tensile Strength of Different polymeric films; (A) *Delonix regia* film without lubricant, (B) *Delonix regia* film with lubricant , (C) HPMC K4M film , (D) Eudragit L100 & S100 mixture (1:2 w/w) film

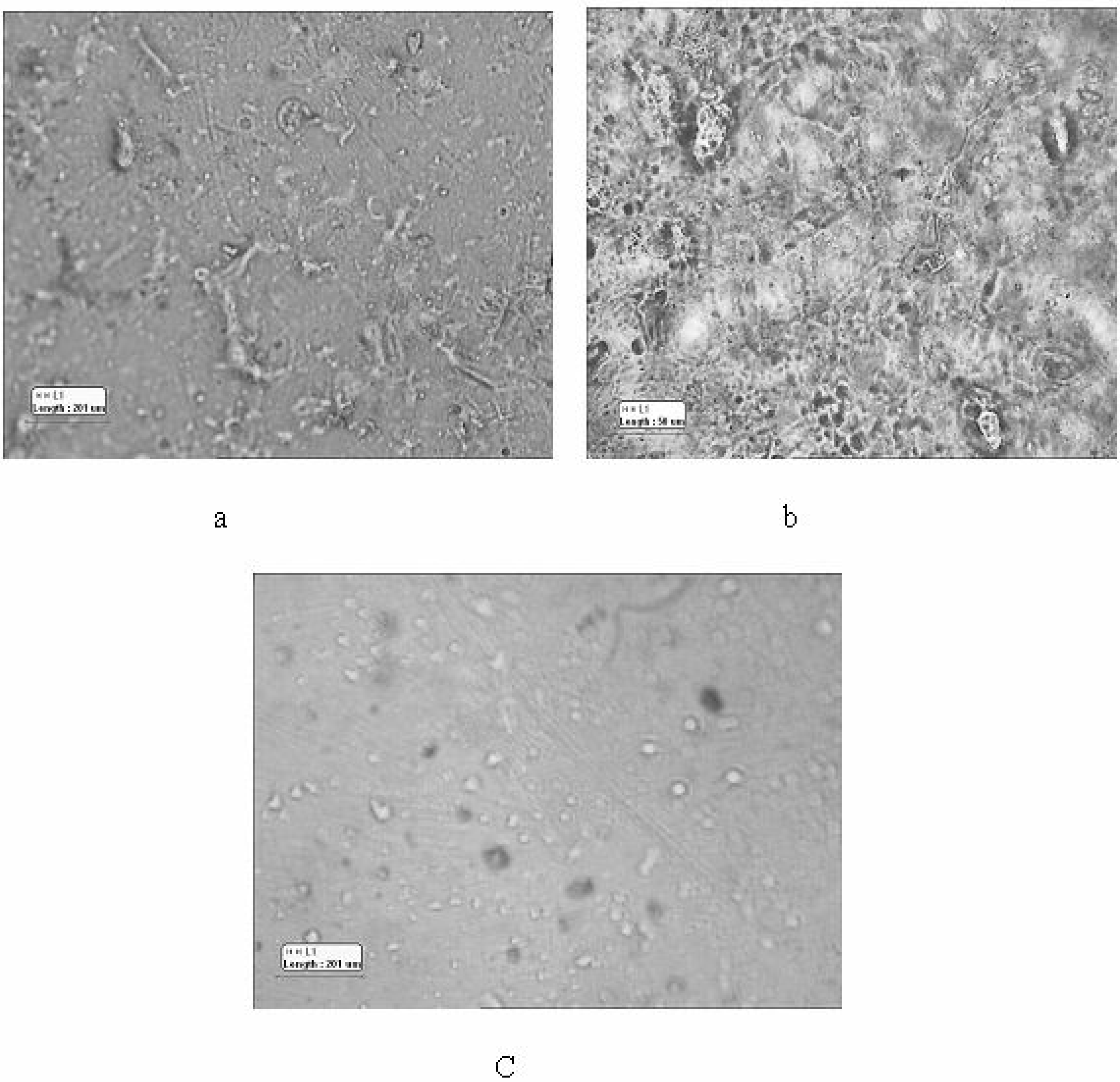

Figure 6. Photomicrographs of prepared polymer films HPMC K 4 M (a), *Delonix regia* (b) and Eudragit S 100 & Eudragit L 100 mixture films (c)

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