Design of Lab Model Mechanical Strength Test Instrument for Tensile Strength Determination of Film Formulations

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Abstract

Objective: The aim of the study was to develop an appropriate and discriminating method to determine the mechanical strength of film formulations by designing and fabricating a lab model mechanical strength test instrument. **Methodology:** Sample films using widely used hydrophilic and hydrophobic polymers were prepared by solvent casting and solvent evaporation method, at three concentrations of 0 2%. Suitable plasticizers were chosen based on the properties of the polymers and the plasticizer concentration 50% w/w depending upon the polymer concentration. **Results:** The prepared films exhibited puncture strength range from 0.89±0.066 N/mm^2 (B1) to 14.73±0.176 N/mm^2 (B4). Films with HPMC K100 and sodium alginate were found to have a higher value for puncture strength and mechanical strength. The developed instrument is suitable for the determination of the tensile strength of various film formulations and can be used as an instructional experimental apparatus to assist students in their efforts to understand the basic mechanical properties of films and related formulations. **Conclusion:** Hydrophilic films were more flexible with a higher value of tensile strength and % elongation at an increasing polymer and plasticizer concentration.

Keywords: *Tensile Strength, Film Formulation, Puncture Strength, Sodium Alginate*

1. INTRODUCTION

The mechanical strength of medicated film formulations is a crucial factor not only during development or production, but also during proper handling by the patient. There are different factors influencing the mechanical properties of films, film forming agent, type and concentration of plasticizer, selection and amount of (residual) solvents, thickness of the final film sheets, type of manufacturing process, storage conditions and the type and amount of active pharmaceutical ingredient in the film. In the development of film formulations, tensile stress-strain relationships are frequently needed. Mechanical strength is an important requirement to ensure damage-free production, packaging, when the product is released to the market and then handling by the patient[1]. The amount of residual solvents and plasticizing agents will significantly influence film flexibility and ability to elongate.[1] To conduct a tensile test, it is first necessary to consider the tensile testing equipment and specimens. The most widely used tensile testing machines are screw-driven testing machines with a moving crosshead and a closed-loop servo-hydraulic testing machine with a hydraulic actuator. However, testing machines are relatively heavy and are typically installed in a laboratory. Conventional test methods for evaluating mechanical properties require a massive testing machine and relatively large material samples.

Objective

The objective of the present study was to develop a mechanical strength test instrument which effectively determines the mechanical strength (tensile strength and % elongation) of film formulations.

2. MATERIAL AND METHODS

Chitosan was procured from Indian Institute of Fisheries, Cochin, India; HPMC-4 KM, Ethyl cellulose, sodium alginate and carbopol 934P were obtained commercially from SD Fine Chemicals. All other reagents and chemicals used were of analytical reagent grade

2.1 Preparation of Films

Both hydrophilic and hydrophobic polymeric films were prepared to evaluate the testing efficacy of the designed instrument. The films were prepared by solvent evaporation technique.[2] Polyethylene glycol and diethyl phthalate were used as plasticizers. Initially a backing membrane of ethyl cellulose solubilised in acetone was prepared onto a glass mould. The film solutions prepared with suitable solubilising solvents was poured over the backing membrane and dried at room temperature. The compositions of the films are as follows**:**

2.2 Development and Fabrication of Mechanical Strength Test Instrument

A tensile strength test apparatus (Figure1) was designed and fabricated at the lab scale. The apparatus consists of a vertical support stand which resides on a metal base plate. The support stand comprises two pulleys which in turn hold a pan on the other side. The pan was tied and supported with metal string. The other side of the string was connected to magnetic holders, one fixed to the base of the apparatus and another connected to the support stand with sliders. The film to be studied should be placed in between the magnetic holders while determining the tensile strength[3]. The weights will be placed in the pan, and the maximum weight required to break the film from the middle is considered as force at break.

2.3 Evaluation Studies

2.3.1 Thickness Determination

The thickness of the films was determined with the help of a screw gauge. Arbitrarily six films from each batch was taken, weighed and calculated the mean.

2.3.2 Weight Determination

Six films from each formulation were collected and weighed in an electronic digital balance.

2.3.3 Viscosity Determination

The viscosity of the film solution was studied using a Brookfield's viscometer (Model DV II + Viscometer). The polymeric dispersion was mixed and transferred into a test tube. The viscosity was determined in the viscometer by using the spindle no.4 at 20 RPM.

2.3.4 Folding Endurance Test-

It was determined by repeatedly folding a small strip of film at the same point till it broke.

2.3.5 Determination of Mechanical Properties of the Films

Films of specified dimensions was used. Mechanical properties of the films were evaluated by the following equations.[1, 2]

Tensile strength (N/mm^2) = force at break (N)/ [width (mm) × thickness of film (mm)]

Elongation at break (%) = (increase in length/original length) × 100

2. RESULTS AND DISCUSSION

The film samples were stored and experiments were conducted under ambient conditions. The time line of the sample measurement was approx. 1-2 min (Taking out the sample from storage, placing into sample holder, measurement start etc.) Therefore, we would assume no immense

change of water content due to sorption or desorption during this short time period.[3] The prepared films exhibited puncture strength range from 0.89±0.066 N/mm^2 (B1) to 14.73±0.176 N/mm^2 (B4). However, higher film thicknesses. Rather, films with more thickness showed lesser puncture strength.

Table 1. Composition of the films

Batch code	Ingredients								
	Chitosan (85%DA) (g)	Eudragit RL 100 (g)	HPMC K100M (g)	Sodium alginate (g)	Sodium CMC (ml)	Guar gum (ml)	HPMC K4M	Diethyl phthalate (% W/W)	PEG 4000 (% W/W)
B1	2	-	-	-	-	-	-	-	50
B2	-	2	-	-	-	-	-	50	-
B3	-	-	2	-	-	-	-	-	50
B4	-	-	-	2	-	-	-	-	50
B5	-	-	-	-	2	-	-	-	50
B6	-	-	-	-	-	2	-	-	50
B7	-	-	-	-	-	-	2	-	50
B8	-	-	-	-	-	-	-	-	50

Table 2. Physical evaluation parameters of films

Batch code	Thickness* (mm)	Weight* (g)	Viscosity* (cps)	Folding endurance#
B1	0.33±0.002	0.098±0.0002	60.87±0.017	>310
B2	0.35±0.003	0.103±0.0005	19.99±0.011	>170
B3	0.20±0.002	0.068±0.0005	28.69±0.011	>280
B4	0.29±0.002	0.067±0.0001	27.77±0.031	>200
B5	0.37±0.001	0.086±0.0003	49.33±0.036	>250
B6	0.48±0.004	0.099±0.0002	77.09±0.035	>300
B7	0.44±0.003	0.101±0.0004	76.39±0.046	>300
B8	0.43±0.004	0.96±0.0003	21.25±0.029	>180

*** Values are represented as mean ±S.D, where (n=5)**

Table 3. Mechanical properties of films

Batch code	Puncture strength* (N/mm^2)	Elongation* (%)	Tensile strength* (N/mm^2)
B1	0.89±0.066	26.73±0.125	0.9963±0.015
B2	10.99±0.366	11.43±0.348	0.2833±0.088
B3	08.99±0.380	36.75±0.235	0.6976±0.062
B4	14.73±0.176	69.25±0.569	1.2282±0.058
B5	6.69±0.341	48.87±0.193	1.2269±0.029
B6	04.72±0.221	38.75±0.148	0.8890±0.063
B7	03.97±0.363	13.72±0.133	0.6323±0.099
B8	01.88±0.212	21.04±0.122	0.6675±0.046

*** Values are represented as mean±S.D, where (n=3), #Average values are represented (n=3)**

Films with highest thickness of 0.48±0.004 mm (B6) showed puncture strength of 04.72±0.221 N/mm^3.

All the prepared films were found to be suitable for the study. Films with HPMC K100 and sodium alginate were found to have a higher value for puncture strength and mechanical strength (Fig 2).

The polymer concentration used for the preparation with a high concentration of plasticizer might have transformed the films into denser. At the same time reduced interpolymer space may have made the films more strong which leads to a higher force per unit area required to break the film.

Films with higher polymer concentration have high viscosity and a higher mechanical strength.[4]

In Fig 2, the relationship between puncture strength and elongation to break has been represented. It was observed that film B4 and B5 prepared with sodium alginate and sodium CMC respectively have high Percent elongation. Being highly moisture absorbing in nature the polymeric films are known to have a high elongation properties.

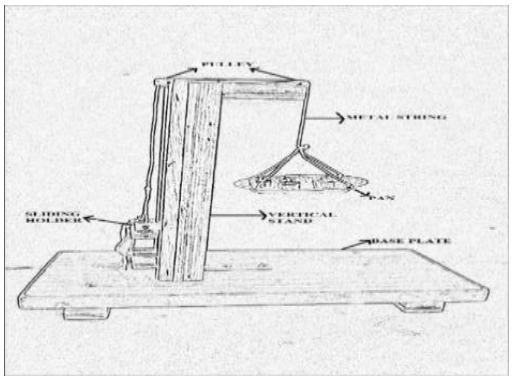

Fig. 1 Mechanical strength test apparatus

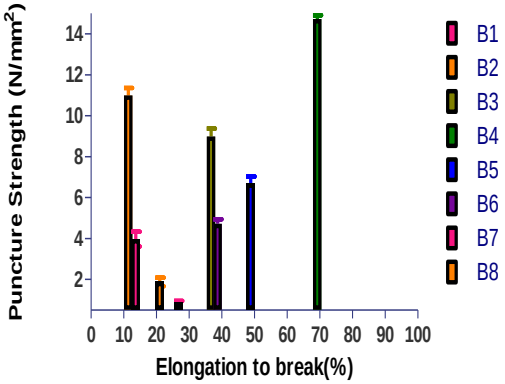

Fig. 2 Stress - strain curve: puncture strength vs. elongation

From the results of the % elongation and tensile strength, it was observed that a higher polymer and plasticizer concentration leads to higher elasticity of the films, which in turn results into a higher tensile strength of the films. Hydrophilic films are more flexible and have a higher value for tensile strength and % elongation with an increasing polymer and plasticizer concentration property.

4. CONCLUSIONS

- The developed instrument is suitable for the determination of the tensile strength of various film formulations.
- The study revealed that it is feasible and suitable method to determine the mechanical strength of film formulations
- The results of the tensile strength can be correlated to the physical, mechanical and stability of the various film dosage forms
- The developed testing system can be used as an instructional experimental apparatus to assist students in their efforts to understand the basic mechanical properties of films and related formulations

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