IJPSR (2019), Volume 10, Issue 10 (Research Article)

INTERNATIONAL JOURNAL OF PHARMACEUTICAL SCIENCES AND RESEARCH

Received on 30 January 2019; received in revised form, 01 August 2019; accepted, 24 September 2019; published 01 October 2019

IMPACT OF HYDROTHERMAL ISOLATION METHOD ON THE RECOVERY OF FENUGREEK SEED HEMI-CELLULOSE ANALYZED THROUGH FTIR-DSC-SEM INTERPRETATIONS

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Keywords:

Fenugreek seeds, Isolation, Hemi-cellulose, FTIR, DSC, SEM, Hydrothermal method, Excipients

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ABSTRACT: Natural Herbal components like seeds mucilages, exudates, gums, polysaccharides, celluloses, hemicelluloses, starch, *etc*. are now a day's exploited as excipients. Literature reveals medicinal, nutraceutical and pharmaceutical uses of fenugreek seed hemicelluloses in dosage form design. Hemicelluloses like galactomannans are water-soluble polysaccharides highly susceptible to hydrolysis either by acidic conditions or by uncontrolled hydrothermal processes. Chances of breakdown of intact polymeric polysaccharides chains into oligosaccharides, disaccharides, monosaccharide, *etc*. increases. Conversion into basic sugar units may also take place leading to changes in the polymeric behavior of the intact structures. So, in the present research percentage hemicellulose recovery is determined by invasive, indirect method in terms of basic glucose units. FTIR-SEM-DSC analysis was found to be useful characterization tools to interpret and support the recovery details of the hemicellulose isolates. Finally, it is concluded that the method of isolation of any natural component either hemicellulose, cellulose, *etc*. targeted to be utilized as excipient entity in dosage form design should be strictly optimized because there are numerous factors in processing which may convert these intact high molecular weight polysaccharide polymeric structures into low molecular weight of monomer units which ultimately may lead to alterations in the functional performance of these structures.

INTRODUCTION: Various useful plants legumes contain mainly glucans (starches and cellulose), hemicelluloses, and sugars as carbohydrates. Many Legume trees exude gums whereas legume seeds additionally have galactomannans and amyloid as endosperm reserve polysaccharides [1]. All these polysaccharides, gums, celluloses, hemicelluloses, starches are now a day's exploited as pharmaceutical excipients.

QUICK RESPONSE CODE

DOI: 10.13040/IJPSR.0975-8232.10(10).4677-84

This article can be accessed online on www.ijpsr.com

DOI link: http://dx.doi.org/10.13040/IJPSR.0975-8232.10(10).4677-84

Moreover, considering the environment concern activity, seed components as pharmaceutical excipients are going to play a very important role in the coming era because it is going to promote the importance of plantation, farming, etc to improve the yield of these natural excipients. The activity is going to promote green thinking, the very important requirement boosting nature-friendly research. Considering the importance of natural seed excipients, the present research work focuses on Fenugreek Seed polysaccharide components.

As such, the health benefits and medicinal properties of Fenugreek Seeds are known since ancient times. Use of Fenugreek seeds in traditional 'Ladoo recipe' is very well known as well.

Fenugreek (*Trigonella foenum-graecum*) is a legume, and it has been used as a spice throughout the world to enhance the sensory quality of foods. It is known for its medicinal qualities such as anti-diabetic, anti-carcinogenic, hypo-cholesterolemic, antioxidant, and immunological activities, *etc*. Besides its medicinal value, it is also used as a part of various food product developments as food stabilizer, adhesive, and emulsifying agent. The medicinal value of fenugreek seeds is mentioned in Ayurveda texts as well as in Greek and Latin pharmacopeia. The seeds are used year-round as a flavoring agent for various dishes. The seeds are also eaten raw as sprouts and used medicinally [2].

The literature also reveals the use of polysaccharide mucilage derived from the seeds of fenugreek as a potential excipient for oral controlled-release matrix tablet [3]. The seeds Polysaccharide mucilage of fenugreek was also investigated as disintegrant for use in mouth dissolving tablet formulations [4, 5]. Literature also reports the use of polysaccharide mucilage as gelling agent [6, 7]. All these dosage forms go through various processes, subjected to various temperature conditions, subjected to stability evaluations. Dosage form stability is evaluated in terms of drug content incorporated into it. Polysaccharide mucilage comes across various experimental parameters. One of the important parameters like temperature could be reason for degradation or breakdown of these polysaccharide components [8].

In comparison to cellulose, hemicelluloses can be branched and sometimes have other than hydroxyl functional groups attached, *i.e.*, carboxyl or acetyl groups. Depending on the sugar composition, level of branching, and the different functional groups, hemicelluloses can have very different properties compared both to cellulose and also other hemicelluloses. Due to the lower molar mass and branching, the hemicelluloses are generally more amorphous than cellulose which makes hydrogen bonding between molecules more difficult.

Therefore, hemicelluloses are normally easier to dissolve in water and more easily hydrolyzed by acid than cellulose. Low temperatures (<100 °C) has been used during extraction to avoid intense hydrolysis and boiling as well as mimic the thermo-mechanical pulping (TMP) process [9].

Natural polysaccharide mucilages should remain intact and stable to be recognized as the acceptable excipients in dosage form design and development. The various experimental parameters can have impact on functional properties of the natural gums, polysaccharides, celluloses, hemicelluloses, etc used as excipient which in turn can have impact on dosage form performance. Because, the natural ingredients are finding the uses as pharmaceutical aid, excipients in modern dosage form development, standardization of these components justifying the quality attributes like identity, safety, efficacy, and purity is the prime focus of the guidelines mentioned for the characterization of excipients [10, 11]. It is very important to consider the impact of these excipients on functional performance of the dosage form. Based on the above understanding the present research work focuses on Impact of hydrothermal isolation method on the recovery of Fenugreek Seed Hemi-cellulose analyzed through FTIR- DSC-SEM interpretations.

Literature reports the breakdown of polysaccharides into glucose or oligosaccharides with the aid of acidic conditions [12]. Along with the stability of the drug, the stability if these excipients are equally important and are one of the important criteria to be taken into consideration. The functional performance of any dosage form is very much based on excipients as well. Any change in excipient during processing, storage, *in-vivo* pH specific changes in the excipient functionality, etc may render the changes in the functionality of the overall dosage form. So, the stability of excipient is equally important in this context. Phenol sulfuric acid method is very well known for the determination of total carbohydrates. Rapid and mass screening method for galactomannan content in fenugreek seeds has been reported in the literature. Total galactomannan and carbohydrate content in different genotypes of fenugreek seeds have been mentioned in the literature. The galactomannan is soluble fiber from fenugreek containing Galactose and mannose units in the ration 1:1. The total carbohydrates are determined in the form of Galactose and mannose units as the working standards. Total galactomannan and carbohydrate contents were determined based on these working standards [13].

Fenugreek seeds mainly contain galactomannan, a water-soluble heteropolysaccharide [14]. Galactomannan is hemicelluloses. Hemicelluloses are characterized as a group of cell wall polysaccharides that are neither cellulose nor pectin and have a β-(1→4)-linked backbones of glucose, mannose or xylose linked in equatorial configuration. Galactomannans present in Fabaceae seeds generally consists of galactose and mannose units, [15] whereas mannose is C2 epimer of Glucose and galactose is C4 epimer [16].

Moreover, Fenugreek seeds are rich in Crude fiber, which is composed of cellulose, which is a complex molecule consisting of glucose molecules. Related to cellulose is hemicellulose - one type of hemicellulose is pectin. Lignin, another form of crude fiber, is not a carbohydrate per se, but it is of plant origin and is also indigestible [17, 18]. As the main component of lignocelluloses, cellulose is a biopolymer consisting of many glucose units connected through β-1,4-glycosidic bonds. Breakage of the β-1,4-glycosidic bonds by acids leads to the hydrolysis of cellulose polymers, resulting in the sugar molecule glucose or oligosaccharides. So any process responsible for isolation of hemicelluloses, celluloses in fenugreek seeds should be controlled in such a way as to avoid hydrolysis and breakdown of the polysaccharide structural background. Mineral acids, such as HCl and H_2SO_4, have been used in the hydrolysis of cellulose. In this context it is thought that the acidic environment at the site of administration that is stomach can be the reason for the hydrolysis of the fenugreek seed hemicelluloses or celluloses.

So, the present research work was planned to study the utilization of phenol sulphuric acid method to indicate the stability of the seed hemicellulose component in case they are explored as the dosage form excipients. Further characterized using FTIR, DSC, and SEM to interpret the conclusions.

MATERIALS AND METHODS:

Isolation of Hemicelluloses from *Trigonella foenum-graecum* Seeds: The Hemicellulose isolates of fenugreek seeds were extracted with Hot water extraction method with slight modification in the methods reported in the literature [9]. Compared to reported method, the temperature was kept low similarly method run time was also reduced. Twenty Grams (20 gm) of fenugreek seeds were dissolved in 400 ml double distilled water and heated at 70 °C for 3 h. With stirring up to slurry formation, it was kept for cooling in refrigerator for 3 to 4 h, for separation of supernatant liquid. The upper clear slurry was decanted and was centrifuged at 500 rpm for 20 min. The supernatant was separated and concentrated at 60 ºC on water bath. The solution was cooled to the room temperature (25 °C) and was poured into thrice the volume of ethanol with continuous stirring. The precipitated material was washed with distilled water and dried at 50-60 ºC. After collection the material was collected and ground to obtain isolated hemicellulose components which were then stored in airtight container. Isolated hemicellulose component is shown in **Fig. 1**.

FITR Determination of Hemicellulose Isolate: The IR spectra of isolated hemicelluloses components was recorded using Fourier Transform Infra-Red Spectrophotometer (SHIMADZU, Japan) with diffuse reflectance principle. A small quantity of isolated powder component was grounded with KBr and then pelletized using KBr press. Scanning was performed between the ranges of 400-4000 cm^{-1}. The Result and interpretation are shown in **Fig. 2** [19].

% Recovery study of Hemicellulose in terms of Basic Glucose Units: [20, 21] Percentage recovery study utilized in the present research work was the invasive indirect techniques to determine the presence of hemicellulose isolates in terms of basic glucose units. Breakdown of long-chain polysaccharide by hydrolyzing it using concentrated acids and reaction of basic glucose units with phenol to give colored dehydrated product hydroxymethyl furfural is the principle behind the method used in the present study. Considering that the lower temperature conditions used during Hot water isolation of Hemicellulose has not hydrolyzed the basic structure or some proportion of polymeric chains might have got hydrolyzed. So, for complete hydrolysis the isolated sample is made to react with concentrated sulphuric acid which in turn is made to react with phenol to give further colored product obtained after the reaction of glucose with phenol.

Materials:

- Phenol 5%: Redistilled (reagent grade) phenol (50g) dissolved in water and diluted to one liter.
- Sulphuric acid 96% reagent grade.
- Standard Glucose: Stock – 100mg in 100 mL of water.
- Working standard – 10mL of stock diluted to 100 ml with distilled water.

Preparation of Blank Solution: To 1 ml of distilled water added 1 ml of 5% phenol followed by 5 ml of concentrated H_2SO_4. This solution was taken as a blank solution.

Preparation of Standard Solution: A stock solution 100µg/ml of glucose was prepared by dissolving 10 mg of glucose in 100 ml distilled water (Stock solution). Aliquots of each 6, 7, 8, 9 and 10 mL were taken from this solution to obtain sugar concentrations 60-100 µg/ml. 1 ml of 5% phenol solution was added to 1 ml of sugar solution followed by 5 ml of concentrated H_2SO_4 (working solution). The absorbance of the working solution was measured after 10 min at 487 nm against blank. The calibration curve for glucose was shown in **Fig. 3**.

Test Preparation: About 10 mg of hemicellulose isolate was dissolved in 100 ml water to get 100 ug/mL (stock solution). Hydrolyzed by adding 5mL of concentrated H_2SO_4. 1 ml of 5% phenol solution was added, and the solution was shaken well. The solution was kept aside for 10 min as the solution becomes very hot due to liberated heat during the hydrolysis process. It is cooled to room temperature. The absorbance of working solution was measured after 10 min at 487 nm against blank. The experiment was carried out in triplicate (*i.e.* Test-1, Test-2 & Test-3) as shown in **Table 1**. In acidic medium, glucose is dehydrated to hydroxymethylfurfural. This forms a green colored product with phenol and has absorption maximum at 490 ± 2 nm. Glucose is estimated in the sample solution using the standard graph [20, 21].

TABLE 1: UV ABSORBANCE FOR STANDARD GLUCOSE AND HEMICELLULOSE TEST ISOLATE

S. no.	Conc. (ug/mL)	Abs. of std.	Abs of test			Mean	%RSD
			Test 1	Test 2	Test 3		
1	60	0.5037					
2	70	0.5536					
3	80	0.5947	0.5621	0.5499	0.5691	0.5603	0.0097
4	90	0.6421					
5	100	0.6745					

Melting Point Determination: The melting point of an isolated mucilage was determined by introducing a tiny amount of mucilage powder into a small capillary tube, attaching this to the stem of a thermometer centered in a heating bath, heated the bath slowly, and observed the temperatures at which melting begins and is completed [22].

Scanning Electron Microscopy (SEM): [23, 24] SEM study was outsourced from the Savitribai Phule Pune University, Pune. SEM study helps to understand the surface morphology of the polymer, images for SEM gives idea about surface morphology of polymer at different sizes. Particle morphology of FGM was examined by SEM (Nova nanosem 450). Samples were analyzed at different magnifications of 5000x by the following procedure. The sample was mounted on the SEM samples tab using a double-sided sticking tape. The samples were coated with Chromium (200A^{o}) under reduced pressure (0.001torr) for 2 min using an ion sputtering device. The chromium coated samples were observed under the SEM and photomicrograph of suitable magnifications was obtained which are shown in **Fig. 5**.

Differential Scanning Calorimetry (DSC): Differential Scanning Calorimetric analysis, or DSC, is a thermal analysis technique that looks at how a material's heat capacity (Cp) is changed by temperature. A sample of known mass is heated or cooled and the changes in its heat capacity are tracked as changes in the heat flow. This allows the detection of transitions such as melts, glass transitions, phase changes, and curing. Because of this flexibility, since most materials exhibit some transitions, DSC is used in many industries, including pharmaceuticals, polymers, food, paper,

printing, manufacturing, agriculture, semi-conductors, and electronics. The biggest advantage of DSC is the ease and speed with which it can be used to see transitions in materials. If you work with polymeric materials of any type, the glass transition is important to understand your material. In liquid crystals, metals, pharmaceuticals, and pure organics, you can see phase changes or polymorphs and study the degree of purity in materials [25, 26, 27].

Procedure: To investigate the change in thermal behavior, DSC analysis was outsourced using Shimadzu DSC-60 (Shimadzu Limited Japan). DSC study of the isolated hemicellulose component was carried out by weighing accurately about 4.0 mg of the powdered component. Samples were heated in an open aluminum pan with reference to an empty sealed pan at a rate of 5 °C/min within a -20° to 300 °C temperature range under a nitrogen flow of 100 ml/min. The result obtained was shown in **Fig. 6**.

RESULT AND DISCUSSION:

Isolation of Mucilage from *Trigonella foenum-graecum* Seeds: As shown in **Fig. 1**, smooth and soft powder was obtained from the isolation of fenugreek seeds. The hemicellulose is water soluble component so expected to get solubilized and swell in the process applied.

FIG. 1: ISOLATED *TRIGONELLA FOENUM-GRAECUM* SEED HEMICELLULOSE COMPONENT

FTIR Determination of Isolated Hemicellulose Component: The characteristics IR spectra's of Hemicelluloses from different sources are referred for interpretation of the data. As shown in **Fig. 2** the group frequencies 3332.99 and 2920.09 cm^{-1} are indicative of fundamental frequencies for -OH stretching and asymmetric vibration of -CH respectively in the hemicellulose structure. These matches with the fundamental group frequencies for glucose which could be the indication of partial hydrolysis of the hemicellulose polysaccharide chain. The group frequency 1026.13 cm^{-1} highlights the C-H bend out of the plane.

As per the reported literature, the characteristic wavelengths around 1380 cm^{-1}, shared by hemicellulose, cellulose, and lignin, correspond to C–H stretching and deformation of $–CH_3$ is found to be slightly shifted with increased percentage transmittance tells about decrease in the dense nature of polysaccharide, might be due to bond breaking or separation of cellulose structure from the hemicellulose during the isolation process [15, 28, 29, 30, 31, 32]. The typical group frequencies for hemicellulose reported in the literature are not prominently observed in the spectra suggests that the method of isolation of any natural component either hemicellulose, cellulose, lignin, pectin *etc*. targeted to be utilized as excipient entity in dosage form design should be strictly optimized because there are numerous factors in processing which may convert these intact high molecular weight polysaccharide polymeric structures into low molecular weight or monomer units which ultimately may lead to alterations in the functional performance of these structures.

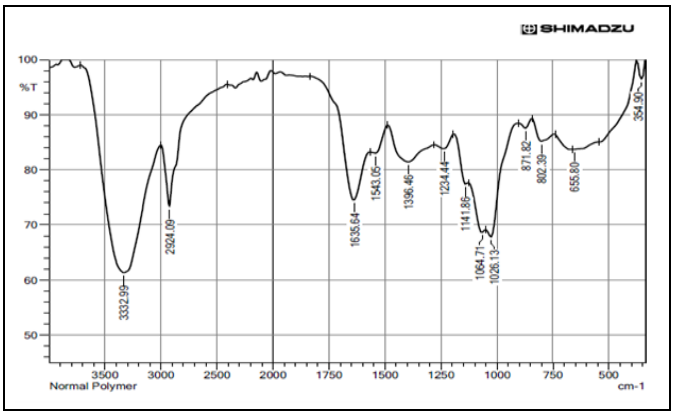

FIG. 2: FT-IR SPECTRA OF HEMICELLULOSE ISOLATE

% Recovery study of Hemicellulose in terms of Basic Glucose Units: Percentage recovery of Isolated Hemicellulose is calculated in terms of Hydrolyzed Glucose units. In hot acidic medium glucose is dehydrated to hydroxymethylfurfural. This forms a green colored product with phenol and has an absorption maximum at 490 ± 2 nm. Percentage recovery calculated using the linear

regression equation (y = 0.0043x + 0.2496) obtained from the standard calibration curve. The standard calibration curve for different concentrations of glucose is represented in **Fig. 3**. A calibration curve was found to be linear in the range of 60-90ug/ml. A correlation coefficient of 0.9956 indicates good linearity between the concentration and absorbance. The % Relative Standard Deviation (% RSD) 0.0097% indicates that the used method is precise & accurate. The total hemicellulose content in terms of basic glucose units was calculated using regression equation obtained from the calibration curve. The hemicellulose content was found to be 72.25% w/w (Mean of three determinations). Isolated hemi-cellulosic components are analyzed spectrophoto-metrically at 487nm **Fig. 4** and percentage recovery was estimated using the standard calibration curve.

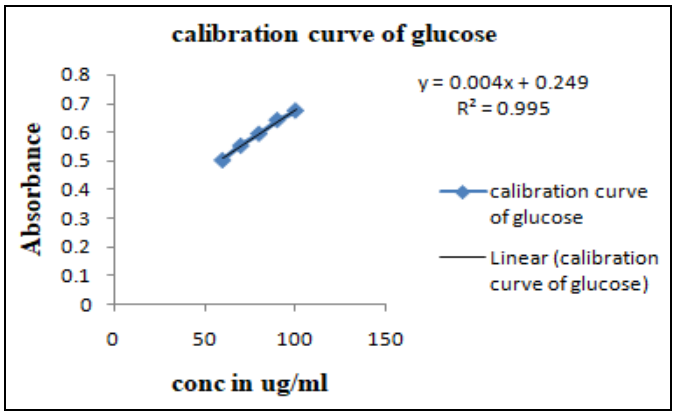

FIG. 3: CALIBRATION CURVE OF GLUCOSE (STANDARD)

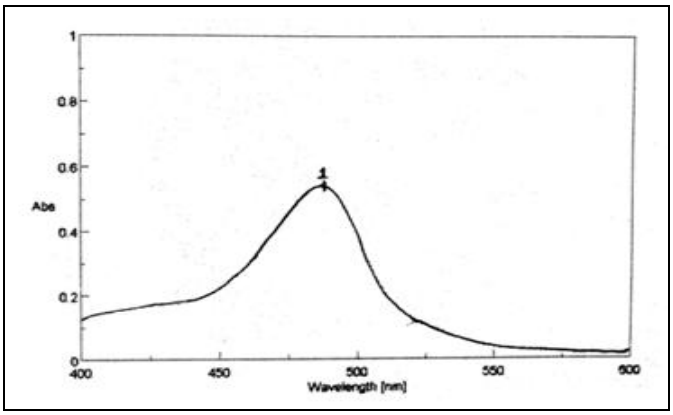

FIG. 4: SPECTRA FOR ISOLATED HEMICELLULOSE COMPONENT

Melting Point of Hemicellulose Isolate: The physical property of a compound, such as melting point can provide useful information which can help in identification of sample or establish its purity. The temperature at which a solid melts and becomes a liquid is the melting point. Since, this requires that the intermolecular forces that hold the solid together have to be overcome, the temperature at which melting occurs will depend on the structure of the molecule involved. A pure, nonionic, crystalline organic compound usually has a sharp and characteristic melting point (usually 0.5-1.0 °C range).

A mixture of very small amounts of miscible impurities will produce a depression of the melting point and an increase in the melting point range. Consequently, the melting point of a compound is a criterion for purity as well as for identification. The hemicellulose isolated sample doesn't melt after applying heat. After heating, above 200 °C it samples charred/burnt which was primary indication of amorphous nature of isolated hemicellulose components.

Scanning Electron Microscopy (SEM) of Hemicellulose Isolate: From **Fig. 5** it was seen that in SEM study, isolated hemicellulose sample shows irregular rough fractures, shape, and size. The loose and compressed organization micrograph are indicative of an amorphous material which supports the melting point determination study. The particles are mostly seen as aggregates of irregular shapes and dimensions which are fibrous in nature. The shape and structure or surface topography of the hemicellulose isolate may be affected by the method of extraction and purification or preparation of the product.

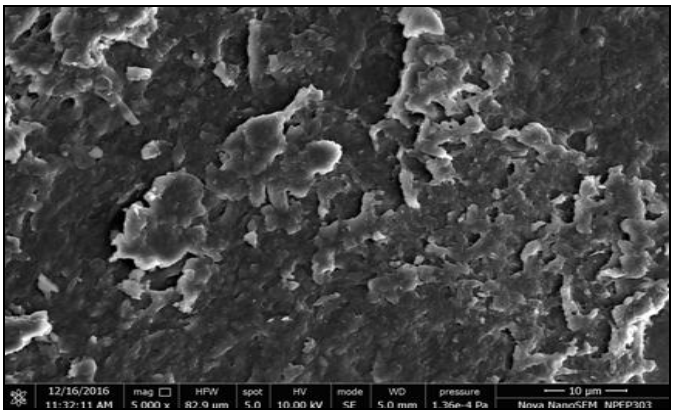

FIG. 5: SCANNING ELECTRON MICROSCOPY (SEM) OF ISOLATED HEMICELLULOSE COMPONENT

Differential Scanning Calorimetry (DSC) of Hemicellulose Isolate: From the DSC thermogram of Hemicellulose isolate, it was observed that glass

transition temperature (T_g) is well below 44.65°C. The point where the mechanical properties of the polymer change from those of an elastic material to those of a brittle one due to changes in chain mobility. Above the glass transition temperature *i.e.* onset of 44.65 °C initiation of exothermic process of crystallization where heat is released to the surroundings can be observed Slight deep (T_C) in the graph could be the indication of crystallization. Complete crystallization is observed at 84.42 °C where exothermic broad peak was seen and endset is observed at 93.70 °C. From the point of temperature of end set initiation of melting phase was observed. Melting is an endothermic process, therefore, absorption of heat can be observed here. The onset of melting, *i.e.* melting process started at 249.02 °C. Complete melting can be observed at endothermic peak 250.84°C. (T_m) End set temperature for melting was found to be 252.28 °C. Note: Study was outsourced. Interpretation is based on the basis of supportive data and the values with the graph. Graphical presentation of peaks seems incomplete in **Fig. 6**.

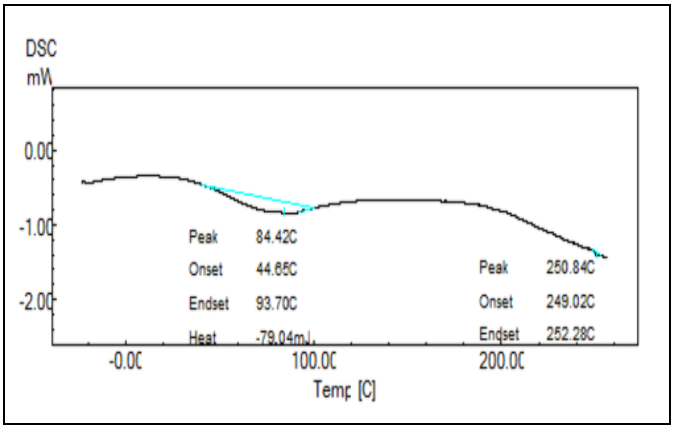

FIG. 6: DSC THERMOGRAM OF ISOLATED HEMICELLULOSE COMPONENT

The data obtained for DSC of Hemicellulose component interprets amorphous behavior of the component. The amorphous portion only undergoes the glass transition while the crystalline regions only undergo melting. The exact temperatures at which the polymer chains undergo these transitions depend on the structure of the polymer. Subtle changes in polymer structure can result in huge changes in Tg. Above the glass transition temperature, the polymer chains have high mobility. At some temperature above Tg the chains have enough energy to form ordered arrangements and undergo crystallization.

Moreover melting endoderm might be an indication of either crystalline transformation of the amorphous hemicellulose structure or possibility of cellulose impurities during the isolation process cannot be denied. If melting endotherm is due to crystalline transformation then the results interpret that the fenugreek seed hemicellulose isolate is very good candidate for the curing. The curing of polymer could be the one more option to take the benefits of functional performance of the cured polymer in dosage form design as pharmaceutical excipient.

CONCLUSION: The specific chemical composition, high imbibitions potential and good adhesive and frictional properties of mucilage envelope are important features that are partially utilized in food technology and medicine. Mucilage is used, *e.g.* as a binding agent or releases retardant in the tablet formulations, as an emulsifying agent in hydrogels and as a lubricant [33]. The loose architecture and special chemical composition of the intact mucilages are important for water binding and storage, which are crucial for the proper functioning of the mucilage envelope.

About the thought, that this natural Mucilage being explored as pharmaceutical aid or excipients playing the role as release retardants, binders, gelling agents, *etc.*, it is necessary to focus on the isolation or preparation method. It is very important to see if the method is invasive or noninvasive. Any invasive method leading to disturbance of structural organization of these mucilaginous compounds which are functionally interrelated with each other may lead to changes in functional performance of the isolate. Maximum possible it should be seen that any natural structure if kept intact may function differently as compared to any changes made in the structure during isolation process.

ACKNOWLEDGEMENT: We would like to thanks the MET's Institute of Pharmacy, Adgoan, Nashik for providing us necessary lab facilities, Grateful thanking for all helping hands, Savitribai Phule Pune University Pune, Maharashtra, India.

CONFLICT OF INTEREST: Nil

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How to cite this article:
Deokar GS, Shewale SS, Kshirsagar SJ and Ahire PD: Impact of hydrothermal isolation method on the recovery of fenugreek seed hemi-cellulose analyzed through FTIR- DSC-SEM interpretations. Int J Pharm Sci & Res 2019; 10(10): 4677-84. doi: 10.13040/IJPSR.0975-8232.10(10).4677-84.

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