

Thermo-gravimetric Analysis of Extracted Methylcellulose from Wheat Straw

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ABSTRACT: Wheat Straw is the agricultural by-product obtained from different parts of wheat plant like stem, leaves, nodes and internodes. Wheat Straw is rich in cellulosic fibers and other elements which make it balanced source for cellulose and cellulosic derivatives methylcellulose. Cellulose was extracted from wheat straw by TAPPI method T-203-cm-99 to inspect their potential for use in formation of derivatives of cellulose. The four different samples of extracted cellulose were used to prepare a methylcellulose and was analyzed by Thermogravimetric analysis (TGA). MBLP, MHMR, MKNG and MMND samples of methylcellulose were prepared by methylation, via iodomethane as alkylating agent respectively. The materials produced were characterized for their thermal properties (TGA). The technique can analyze materials that exhibit either mass loss or gain due to decomposition of volatile component such as moisture. TGA curves reveal the weight loss percentage of materials with respect to the temperature of thermal degradation. TGA technique has used to check the thermal stability of prepared methylcellulose and the results were compared with the Standard Methylcellulose (SM) sample (Methylcellulose, Central Drug House (p) Ltd. New Delhi). All extracted methylcellulose samples displayed good thermal stability with SM.

Keywords: Cellulose; iodomethane; methylcellulose; wheat straw

INTRODUCTION

We utilize agricultural raw material, wheat straw for the extraction of methylcellulose because wheat is grown-up in most of regions in India and rich in cellulosic products. Wheat straw is primarily composed of cellulose, hemicellulose and lignin [1]. More than 600 million tons wheat straw is produced yearly whole of the world, most of wheat straw are charred in the ground convey major environmental and safety hazards [2, 3]. Being rich in carbohydrates like cellulose, hemicelluloses, proteins, minerals, silica and fiery debris, their cell divider is very rich in cellulose filaments that were appropriate for examination of methylcellulose [4].

Cellulose is a most abundant polymers which have known to have high potential applications in various fields [5, 6] Cellulose is a polysaccharide, extensive with hundreds or thousands of glucose particles, β -(1 \rightarrow 4)-connected D-glucopyranose units with three hydroxyl groups, which having shape complex between inter- and intra-atomic hydrogen bonds [7]. Cellulose can't be broken up in similar way to solvents and does not soften prior to the thermal degradation. Various activities have been made to be familiar with realistic solvents for cellulose [8]. In contrast, the cellulose molecule is a very long polymer of glucose units, and its crystalline regions improve the thermal stability of wood [9]. Wheat straw extracted cellulose which is unsolvable in organic solvents and water. In this manner, cellulose is often changed over into its

derivatives methylcellulose. Methylcellulose is a cellulose derivative that can be set up from the reaction of soluble base cellulose with dimethyl sulphate or iodomethane [10-12]. It may be used as thickener, drugs in pharmaceutical industry, food, civil construction and as an administrator for adjusting water thickness in the petrochemical business for considerable oil recuperation which empower business [13-18]. The methylcellulose prepared with methylating reagent, were characterized by Thermo-gravimetric Analysis (TGA) by chemical method.

MATERIALS AND METHODS

Sampling Sites: Wheat straw was taken from local agricultural area of Himachal Pradesh of four different districts Bilaspur (BLP), Hamirpur (HMR), Kangra (KNG) and Mandi (MND), India. These samples taken from Himachal Pradesh by the following reasons like very less use other than animal feeding, burning of wheat straw in the field which decreases the soil fertility and also not managed properly. After drying in sunlight, it was ground and sieved under mesh screens. Wheat Straw was dried in oven at 105 °C for 3hrs and stored at room temperature in air tight container. Reagent used iodomethane, ethanol, acetic acid, sodium chlorite and acetone of analytical grade was used in the experiment. Distilled water was used throughout the experiment.

Extraction of Cellulose and Synthesis of Methylcellulose: Cellulose was extracted from wheat straw

of different sampling sites as Bilaspur (CBLP), Hamirpur (CHMR), Kangra (CKNG) and Mandi (CMND) by TAPPI method, T-203-cm-99 [19]. 5gm holocellulose was prepared from oven dry dust which is obtained from wheat straw. It was treated with 30ml of 17.5 percent NaOH at 20 °C. After standing for 5mins duration with 10ml portions with steady rousing, the sample mixture is macerated with flattened glass rod. After 30 mins, 75 ml of uncontaminated water was added at 20 °C with stirring and then the materials was acceptable to place for 30 mins 100 ml of pure water at 20 °C was added again and the contents were kept for 30 mins more in contact with alkali. The remains was filtered and then soaked in 8.3% NaOH for few minutes and drained by suction. The residue was rinse with 250 ml of pure water and saturated in 2 N acetic acid for 5mins In conclusion mixture was rinse with 400ml of pure water and dehydrated in oven at 105±1 °C. The alpha-cellulose content was determined on O.D. basis as:

$$\text{Percentage of alpha cellulose content} = \frac{w \times 100}{W}$$

Where; w = weight in gram of residue and W = weight in gram of holocellulose taken for test.

Methylcelluloses [Bilaspur (MBLP), Hamirpur (MHMR), Kangra (MKNG) and Mandi (MMND)] were synthesized from different extracted cellulose [CBLP, CHMR, CKNG and CMND] by methylation process. 5 gm α-cellulose oven dry weight was mercerized in 100 grams 40 % sodium hydroxide solution for 1hr at the room temperature. Upon the filtration, the mercerized pulp, 150ml 2-propanol, and 50ml iodomethane were added into a flask. The methylation reaction lasted for 22 hrs at 60 °C. This process was continual. Finally, Methylcellulose samples were saving in a refrigerator at 4 °C [20].

Characterization of Methylcelluloses: TGA:TGA arc were acquire in a NETZSCH STA 409 C/CD using aluminum and DTA/TG crucible alumina pot as indication. The research was agreed out under constant nitrogen run of 50mLmin⁻¹, and the temperature ramp was set at 55/10°C min⁻¹. A 10mg sample was utilized and the mass decreases were recorded from room temperature to 500 °C. A TGA spectrum of standard methylcellulose was compared with the spectra of prepared Methylcellulose.

RESULTS AND DISCUSSION

TGA thermo-gram of SM and treated MBLP, MMND, MKNG and MHMR are illustrated in Figures 1-5. The representative TGA thermo-gram of SM showed thermal degradation. The thermal degradation SM commenced at around 54.7 °C and terminated at around 500 °C. During this thermal process, the SM

lost 71.79 % of its original weight during this event. The SM sample had lost 71.79 and 4.85 % in the first and second step of degradation, respectively of their total original weight during this process in figure. The major weight loss was in the first step of degradation 71.79 %, which occurred in the temperature range of 54.7–380 °C, which was complied with the TGA thermo-gram. The residual mass of SM is 17.91 % at temperature 500 °C.

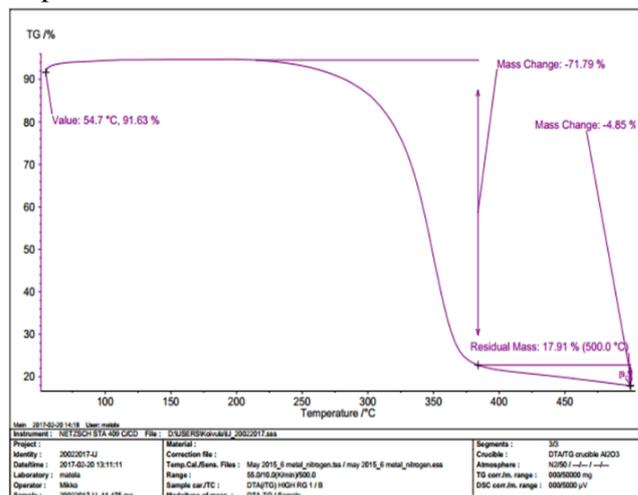


Figure 1: Thermo-gravimetric Analysis (TGA) of Standard Methylcellulose (SM).

The thermal degradation MBLP commenced at around 55.3 °C and terminated at around 500 °C. During this thermal process, the MBLP lost 68.46 % of its original weight during this event. The MBLP sample had lost 68.46 and 4.57 % in the first and second step of degradation, respectively of their total original weight during this process in figure. The major weight loss was in the first step of degradation 68.46 %, which occurred in the temperature range of 55.3–380 °C, which was complied with the TGA thermo-gram. The residual mass of MBLP is 24.31% at temperature 500 °C.

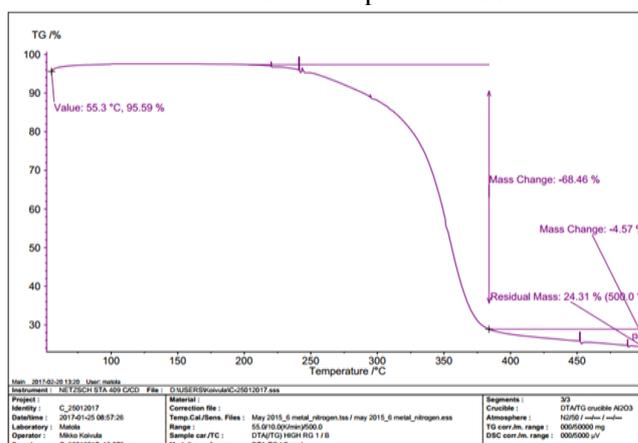


Figure 2: Thermo-gravimetric Analysis (TGA) of prepared Methylcellulose from Bilaspur District Wheat Straw (MBLP).

The thermal degradation MHMR commenced at around 55.1 °C and terminated at around 500 °C. During this thermal process, the MHMR lost 68.12 % of its original weight during this event. The MHMR sample had lost 33.17, 29.58 and 5.44 % in the first, second and third step of degradation, respectively of their total original weight during this process in figure. The major weight loss was in the first and second step of degradation 33.17 and 29.58 %, which occurred in the temperature range of 55.1–375 °C, which was complied with the TGA thermo-gram. The residual mass of MHMR is 26.03 % at temperature 500 °C.

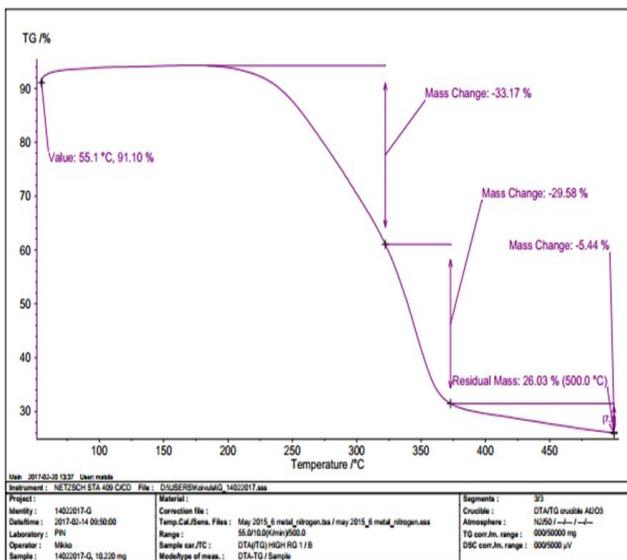


Figure 3: Thermo-gravimetric Analysis (TGA) of prepared Methylcellulose from Hamirpur District Wheat Straw (MHMR).

The treated MHMR sample showed a considerable same in Temperature (375 °C) as compared to the SM sample. The comparative evaluation of the maximum temperature showed same in thermal stability of treated MHMR as compared to SM.

The thermal degradation MKNG commenced at around 55.4 °C and terminated at around 500 °C. During this thermal process, the MKNG lost 68.12 % of its original weight during this event. The MKNG sample had lost 68.12 and 5.89 % in the first and second step of degradation, respectively of their total original weight during this process in figure. The major weight loss was in the first step of degradation 68.12 %, which occurred in the temperature range of 55.4–375 °C, which was complied with the TGA thermo-gram. The residual mass of MKNG is 22.58 % at temperature 500 °C.

The thermal degradation MMND commenced at around 55.7 °C and terminated at around 500 °C. During this thermal process, the MMND lost 72.45 % of

its original weight during this event. The MMND sample had lost 72.45 and 4.22 % in the first and second step of degradation, respectively of their total original weight during this process in figure. The major weight loss was in the first step of degradation 72.45 %, which occurred in the temperature range of 55.7–380 °C, which was complied with the TGA thermo-gram. The residual mass of MMND is 19.74 % at temperature 500 °C.

The treated MMND sample showed a considerable same in Temperature (380 °C) as compared to the SM sample. The comparative evaluation of the maximum temperature showed same in thermal stability of treated MBLP and MMND as compared to SM.

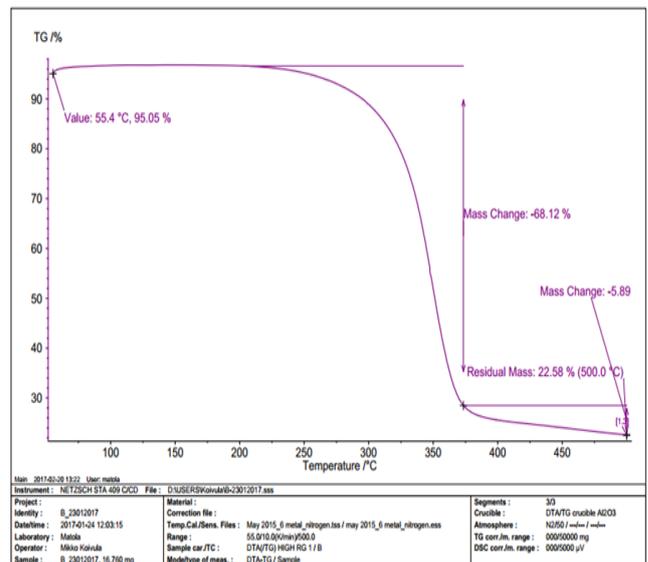


Figure 4: Thermo-gravimetric Analysis (TGA) of prepared Methylcellulose from Kangra District Wheat Straw (MKNG).

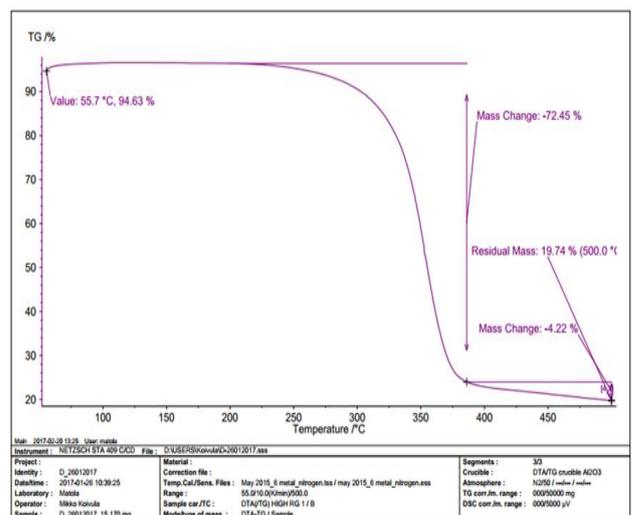


Figure 5: Thermo-gravimetric Analysis (TGA) of prepared Methylcellulose from Mandi District Wheat Straw (MMND)

Table 1: Thermo-gravimetric analysis (TGA) of standard methylcellulose and prepared Methylcellulose from Wheat Straw.

Sample/ parameters	Temp. °C (TG/ %)	Mass change (%)	Residue mass % (temp. °C)
SM	54.7 (91.63)	76.64	17.71 (500)
MBLP	55.3 (95.59)	73.03	24.31 (500)
MHMR	55.1 (91.10)	68.19	26.03 (500)
MKNG	55.4 (95.05)	74.01	22.58 (500)
MMND	55.7 (94.63)	76.67	19.74 (500)

CONCLUSIONS

TGA measures the amount of change in the mass of a sample as a function of temperature or time in a controlled atmosphere. The measurements are used primarily to determine the thermal stabilities of materials as well as their compositional properties. The main decomposition step occurs in the approximately range of 220 °C to 500 °C for methylcellulose samples. In stage one, the initial weight loss step occurred at 50–200 °C due to the evaporation of water absorbed. Stage two is seen to take place around 250–350 °C and is attributed to the degradation of components of carbohydrates in the cellulose samples, which are converted to volatile gases such as CO, CO₂, and CH₄. The final stage of degradation occurred over a wide range of temperatures above 350 °C. Within this stage, degraded volatile products are getting removed. Furthermore, the chemical modification of cellulose leads to significant structural changes which impact on the thermal properties of the produced cellulose derivative methylcellulose. Since that methylcellulose decomposition occurs in a single step from 220.0 to 410 °C, as discussed above in figures TG curves. When compared with the standard Methylcelluloses with extracted methylcellulose from wheat straw. All samples showed degradation temperatures around 350 °C. The main weight loss is observed above 300 °C. The stages shown in the TG curves generally involve dehydration (25-100 °C), depolymerisation and pyrolytic decomposition (220-400 °C). Methylcellulose samples showed a smaller weight loss than hydrolyzed cellulose sample in this stage. The comparison of thermo-gravimetric curves for samples of methylcellulose and standard methylcellulose shows a small displacement of the curves to higher temperatures. This aspect

shows that the thermal stability of methylcellulose sample is equal that observed for standard methylcellulose.

REFERENCES

1. Yasin M., Bhutto A.W., Bazmi A.A. and Karim S. (2010) Efficient Utilization of Rice-Wheat Straw to Produce Value Added Composite Products. *Int. J. Chem. Env. Eng.* 1: 136-143.
2. Kumar S. and Walia Y.K. (2014) Harnessing Economical Potential of Methylcellulose from Wheat Straw. *Asian J. of Adv. Basic Sci.* 2: 12-22.
3. Kumar S. and Walia Y. K. (2017) Extraction of Methylcellulose from Wheat Straw of Himachal Pradesh, India. *Orient. J. Chem.* 33: 2625-2631.
4. Mckean W.T. and Jacobs R.S. (1997) Wheat Straw as a Study Fiber Source, Tech. Rep. Recycling Technology Assistance Partnership, Clean Washington Centre, Seattle, Washington.
5. Schurz J. (1999) Trends in Polymer Science: A Bright Future for Cellulose. *Prog. Polym. Sci.* 24: 481-483.
6. Sun X.F., Sun R.C., Su Y. and Sun J.X. (2004) Comparative Study of Crude and Purified Cellulose from Wheat Straw. *J. Agric Food Chem.* 52: 839-847.
7. Klemm D., Heublein B., Fink H. P. and Bohn A. (2005) Cellulose Fascinating Biopolymer and Sustainable Raw Material. *Angew. Chem. Int. Ed.* 44: 33-58.
8. Jin H.J., Zha C.X. and Gu L.X. (2007) Direct Dissolution of Cellulose in NaOH/Thio-Urea/Urea Aqueous Solution. *Carbohydr Res.* 342: 851-858.
9. Yang H., Yan R., Chen H., Zheng C., Lee D. H. and Liang D.T. (2006) In-depth Investigation of Biomass Pyrolysis Based on three Major Components: Hemicellulose, Cellulose and Lignin. *Energy Fuels* 20: 388–393.
10. Ott E. (1943) High Polymers Cellulose and Cellulose Derivatives. New York: Interscience Publishers Inc.
11. Mansour O.Y., Nagaty A. and El-Zawawy W. K. (1994) Variables Affecting the Methylation Reactions of Cellulose. *J. Applied Polymer Sci.* 54: 519-524.
12. Ye D.Y. and Farriol X. (2007) Preparation and Characterization of Methylcellulose from

- some Annual Plants. *Ind. Crops Prod.* 26: 54-62.
13. Jonas R. and Farah L.F. (1998) Production and Application of Microbial Cellulose. *Polym. Degrad. Stab.* 59: 101-106.
 14. Mitchel K., Ford J.L., Armstrong D. J., Elliot P.N.C., Hogan J. E. and Rostron C. (1993) The Influence of Substitution type on the Performance of Methylcellulose and Hydroxypropyl Methylcellulose in Gels and Matrices. *International J. Pharm.* 100: 143-154.
 15. Fu X. and Chung D.D.L. (1996) Effect of Methylcellulose Admixture on the Mechanical Properties of Cement. *Cement and Concrete Res.* 26: 1007-1012.
 16. Borchardt J.K. (1991) Viscosity Behavior and Oil-Recovery Properties of Interacting Polymers. *ACS Symp Ser.* 467: 446-465.
 17. Vieira R.G.P., Rodrigues Filho G., Assuncao R.M.N., Meireles C.S., Vieira J.G. and Oliveira G.S. (2007) Synthesis and Characterization of Methylcellulose from Sugar cane bagasse Cellulose. *Carbohydr. Polym.* 67: 182-189.
 18. Xue J. and Ngadi M. (2009) Effects of Methylcellulose, Xanthan Gum and Carboxymethylcellulose on thermal Properties of better Systems Formulated with different Flour Combinations. *Food Hydrocolloid* 23: 286-295.
 19. TAPPI (US Technical Association of Pulp and Paper Industry), norm T 203 cm 99, (1999).
 20. Ye D. Y. and Farriol Y. (2005) A Facial Method to Prepare Methylcellulose from Annual Plants using Iodomethane. *E. Polymers* 41: 1-13.