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Thermal Analysis of Chitosan by using Differential Scanning Calorimetry

Ramesh Jamre
P.G. Research Scholar
Department of Mechanical Engineering,
Oriental University
Indore, (M.P.) [INDIA]
Email: jamre777@gmail.com

Mohit Maheshwarkar
Associate Professor & Head of the Department
Department of Mechanical Engineering,
Oriental University
Indore (M.P.), [INDIA]
Email: mohitmaheshwarkar@gmail.com

Abstract*—The present paper describes the investigation of thermal properties of chitosan. Thermal properties of the chitosan demonstrated that are able to support the predictability of fire performance data, thermal stability and flammability also. Government concern over the environment, the depletion of fossil fuels and climate change has promoted the development of biopolymers. Compared to polymeric resources from petroleum, natural polymers from renewable resources have the advantages of biodegradability, biocompatibility, non-toxicity, high reactivity, low cost and ease of availability.). dsc curve of chitosan was appeared the curve demonstrates that weight reduction happens in two phases. First stage begins around 85.44oC and the second stage begins around 309.60oC. the primary stage is appointed to the loss of water since polysaccharides more often than not have a solid proclivity for water and in this way might be effectively hydrated.*

The second one compares to the thermal degradation of chitosn. the decomposition temperature of chitosan was observed to be 309.60°C. this outcome.

Keywords:— *Biopolymer, chitin, chitosan, fire performance, thermal properties, crab shell.*

1. INTRODUCTION

The chitosan discovered was in 1859 by Rouget here chitin was subjected to treatment solutions with potassium hydroxide. During the period of 1894,Gilson confirmed the glucosamine is present in chitin and in same time duration was named chitosan by Hopper Seyler (7). since then a lot of researches show with interests in the field of chitin application and aiming the knowledge about this structural relates and properties of polysaccharides and its derivatives also. Chitin deacetylated form known main derivative is chitosan which percentage free NH2 group is greater than 60% it is represented the degree of deacetylation. chitosan is measure of the average number of 2- acetamide-2-dexosi-D-glucopyranose and 2 -amino-2-deoxy-D-glucopyranose units represents the average degree of acetylation. Here solubility of chitosan direct influence the relative percentage of these unit and is an important parameter to determine the degree of acetylation direct or indirectly the average deacetylation degree which from this way free amino group concentration represents. Chitin acetamido groups is low reactive as compare to the amino groups of chitosan. In amino group groups the free electron pair of nitrogen is responsible for the adsorption of metallic cations. The fraction of amino groups that are available for interaction with metals determines by the average degree of

deacetylation. Chitosan is soluble in bellow given dilute acids for example as lactic acid, formic acid, acetic acid as well as inorganic acids. however the solubility is dependent on a lot of parameters such as the molar mass, concentration of acid, degree of deacetylation, and biopolymer ionic strength. Due to beneficial properties or characteristics of biodegrasability, biocompatibility, antibacterial properties bioactivity and this is processed in various forms.

1.1 Objective of the Research

Following are the objectives of the research:

To use the Narmada riverside crab shell squander- Narmada is extremely long stream streams in Madhya Pradesh and Gujarat conditions of India. It is a spotless water waterway having hug crab riverside. Here the crab shells are simply waste material which pollutes environment. These waste materials are abundantly available at riversides and fish market concerns of the Narmada riverside towns, towns and urban areas. To acquire chitin and chitosan biopolymer from these waste crab shells not only save the environment as well as would supplant the plastic materials. Consequently condition and practical issues can be overseen. So this is one of the significant goals of the present study.

To build up a novel biopolymer from Narmada riverside crab shells-these waste crab shells can be used to get ready chitin and chitosan. The extraction of chitin should be possible by compound and natural strategies. The crab shell contains chitin, protein and minerals. The deproteinization and demineralization are the primary procedures to remove chitin from crab shells. The deacetylation of chitin is done to set up the chitosan which is the significant biopolymer to be utilized generally in biomedical applications. The chitosan has great film shaping ability thus it can supplant the polythene packs from the market. So, to build up the chitin and chitosan from Narmada riverside crab shells by compound strategy is the second significant targets of the present investigation.

To research the thermal properties of the chitosan-It is important to understand about the warm properties of arranged chitosan (biopolymer). The chitin and chitosan so created can be used in numerous applications yet there is one significant limitation in its usage since research and examination the thermal properties of chitin and chitosan is unknown of this material. The chitosan can be set up to investigate the thermal properties. For the most part the itemized degree of predictibility of flame execution information, Its importance should not be underestimated; chitin and chitosan (biopolymer) can not burn if they do not break down.

2. LITERATURE REVIEW

Following are the details of research contributions from different researchers in the field of Differential Scanning Calorimetry of chitosan.

2.1 Research contributions from Researchers

1) Jana s.et al.[2017]

According to researcher, DSC is a significant device for the assurance of glass change and softening conduct of polymers. Figure 2.1 (control CS T1 and CS T2 demonstrates the DSC thermo grams of CS. DSC thermo gram of CS test did not show any glass progress temperature which was chiefly connected with its inflexible crystalline nature and event of solid intra sub-atomic hydrogen holding. DSC thermo grams of control CS displayed an expansive exothermic top at 297^{o}C. this exothermic pinnacle can be connected to decay of amine units of the CS test. DSC thermo grams of treated CS T1 test appeared (figure 2.1) an expansion in exothermic temperature pinnacle and it was seen at 325^{o}C. this demonstrated the late disintegration of the amine units of CS T1 as for control. DSC thermo grams of CS T2 demonstrated an exothermic crest at 300^{o}C the expansion in exothermic disintegration temperature could be related

with solid hydrogen bond development after bio field treatment in chitosan.

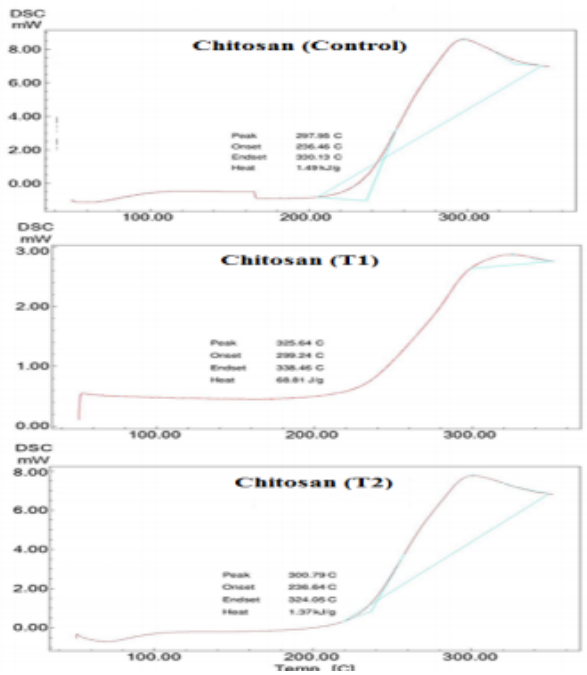

Figure 2.1: DSC Thermo Gram of Control and Treated Chitosan (T1 and T2)

2) Akhilesh Vikram Singh [2013]

the present examination worry with thermal investigation of biomaterial, for example, Chitosan, the DSC bend of the regular polymers demonstrated its particular endothermic pinnacles and change in Hf esteems, and useful in deciding the wellness of the polymers with dynamic medication. The medication and normal polymer similarity study is currently one of the perceived techniques for preformulation venture in pharmaceutical medication advancement. All the characteristic polymers indicated endothermic crest underneath 150°C, and this make them reasonable with a wide class of restorative medications. demonstrates an average DSC bend of chitosan displaying its To (50.42°C) and Tp (106.41°C).

3) P. Dhawade et al. [2012]

In this research studied in differential scanning calorimetry (DSC), leftover dampness misfortune in tests regularly covers and twists warm occasions, for example, glass changes. Temperature tweaked DSC (TMDSC) is isolating such covering procedures. In this investigation chitosan was depolymerised by oxidative corruption technique. Chito-oligomers of various sub-atomic loads were created by this strategy weaked DSC. TMDSC diagrams of chitosan and its oligomers are outlined in figure 2.2.(a-d). decision of skillet during TMDSC estimation is significant. along these lines, the investigation of temperature adjuste fixed dish so as to stay away from the loss of water present in them. From customary DSC the Tg of glass progress temp. of chitosan has showed up at 118 °C in dry state. In nearness of water and by temperature balanced DSC investigation, it turned out to be at 61 °C. this affirm water deos goes about as demonstrates same sort of thermographs for complete warmth stream and non – switching warmth stream. Because of this Tg of the material can't be explained, as the nearness of water is covering the Tg. in any case, when the all out warmth stream is additionally separated into turning around warmth stream and non-switching warmth stream, the Tg turns out to be more prominent.in switching warmth stream Tg for chitosan is acquired at 61.37 °C. the water which is displaying in three unique structures stsrts freeing at different stages. Solidifying water with a softening point underneath that of liquefying purpose of water turns out at around-5 °C, firmly bound water discharges in the scope of 10°C. 100°C and free approximately bound water is freed at about 150°C

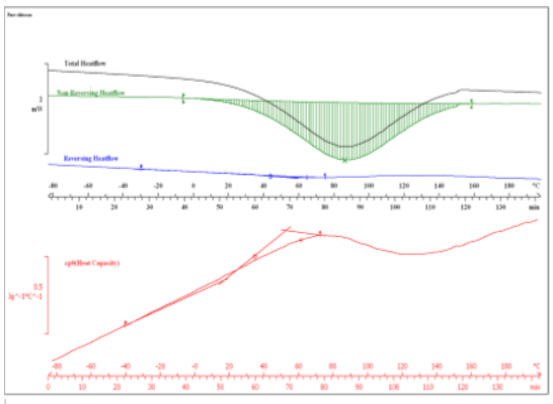

Figure-2.2:.a Modulated DSC TOPEM® of pure chitosan (B)

4) Sukurai et al.(2000)

According to researcher, There are a couple of interchanges concerning the unwinding temperature relating to the glass change temperature of chitosan. We prevailing with regards to watching the tg of chitosan (to be ca. 203 °C) by the immediate and cautious estimation of differential checking calorimetry (DSC), which had been expected not to be touchy enough to identify it. Notwithstanding this perception, the DMA estimation in the second warming run demonstrated the - unwinding at a similar temperature as the glass change temperature saw in DSC. Differential scanning calorimetry (DSC) estimations on Perkin Elmer DSC7. DSC bend of each film were acquired from the second warming keep running at a rate of 10K/min, after the main keep running of warming up to 190 °C and cooling to 25 °C at a similar rate 10K/min, under nitrogen environment, so as to gauge the glass change temperature.

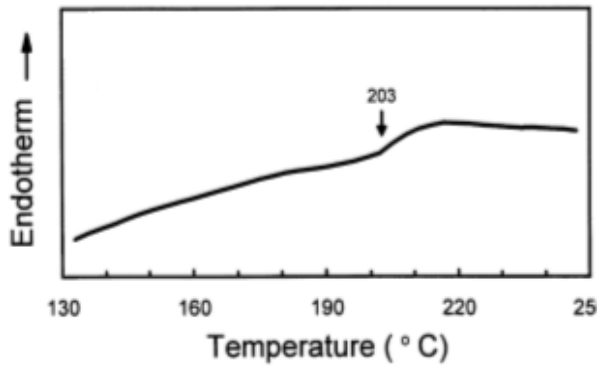

Figure 2.3. DSC Thermogram of Chitosan in the Second Heating Rum.

5) Faridah M Yusoh and Zakaria (2018),

impact of level of dracetylation of chitsosan on concoction structure and similarity of chitosan/polyamide 6 mixes. arrangement mixing method was utilized to plan chitosan/polyamide 6 (PA6) mixes, where the two mixes contain appropriate practical gatherings that can be required to advance similarity between the polymers polymer and its mix were analyzed by DSC investigation. in this, softening (Tm) and crystallization (Tc) change are accounted for as the most extreme and least pinnacle statures respectively.Glass progress temperature (Tg) is accounted for as the midpoint of the base – line of irregularity. Figure 5 demonstrates the thermogram of unadulterated chitosan film which shows sharp exothermic pead at 100 °C, credited by water misfortune. Another expansive endothermic top at 270.7 °C is demonstrating deterioration of chitosan film[60]. As detailed in the past examination, warm disintegration temperature of chitosan was higher than 250.C and it was viewed as that chitosan did not demonstrate a glass change before the deterioration temperature. while figure-2.4 is the thermogram of PA6 which demonstrates a solitary, very much settled endothermic top at 220 °C. Glass progress temperatures (Tg) not watched for the PA6. The thermograms of all CN/PA6 mixes (figure 7-10) demonstrates that the corruption example of all the film are unique and they have either more quantities of debasement top or have diverse vitality esteems for corruption. For thermogram of CN85/PA6 mix an exothermic pinnacle can be seen at scope of 100 °C, relating to the lack of hydration because of loss of water atoms which is unequivocally caught up in the examples. The examples additionally show crests at 216.5 °C, which came about because of the best thermal corruption of the examples

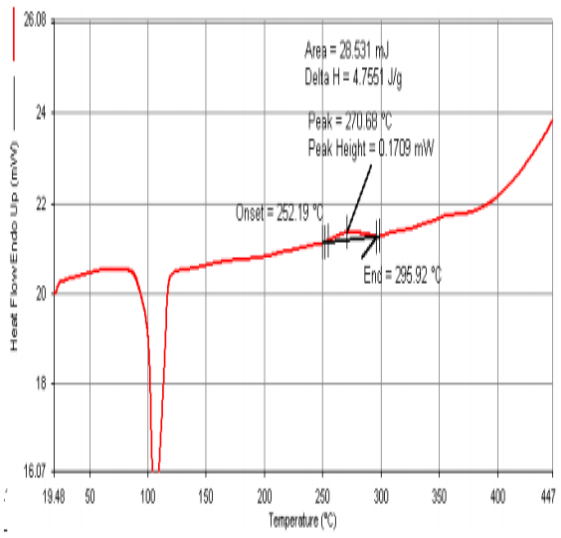

Figure 2.4: DSC Thermograms of pure Chitosan

6) Liao and Hung[2004]

This paper provides that did a motor investigation of warm debasements of chitosan/polycaprolactam mixes. They considered the warm corruption of chitosan mixed with PA6 by thermogravimetric examination and motor investigation (by the ozawa technique). Dry chitosan and PA6 showed a solitary phase of warm debasement and chitosan/PA6 mixes having>20 wt% PA6 displayed in any event two phases of corruption. Figure 2.5(a) demonstrates DSC sweep of chitosan, an endothermic pinnacle based on 100 °C can be doled out to the vanishing of adsorbed water. The example after that began DSC examine. We didn't locate the endothermic top on DSC bend (as appeared in figure 2.5(b), the DSC bend of chitosan does not demonstrate any endothermic progress between room allocates the absence of any crystalline or some other stage change during the warming procedure. Sreenivasan recommended that the solid endothermic crest around 270°C was because of the oxidative debasement of chitosan. Subsequently the endothermic pinnacle revolved around 287°C in figure 2(b) should the warm corruption of the example.

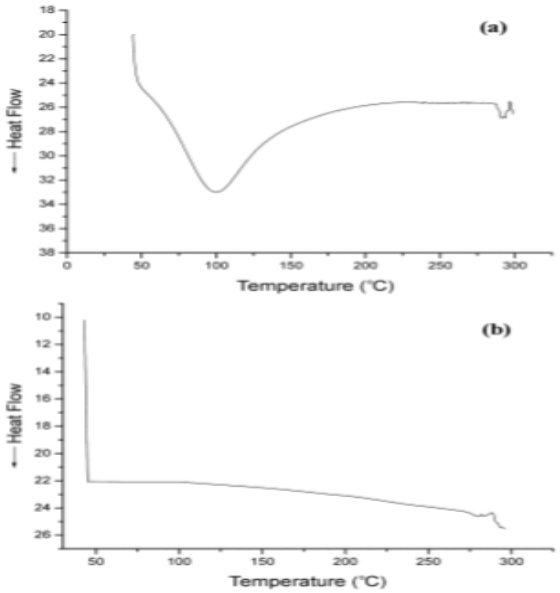

Figure 2.5.(a) DSCcurve of chitosan acquired at a warming rate of 10°C /min and (b) DSC bend of dry chitos*an got at a warming rate of 10°C* /min in an environment of nitrogen,

2.2 Gaps in the research and objectives of proposed research

On the basis of literature survey, following research gaps are being investigated.

1. There is very limited research based on Investigation of Thermal Properties of chitosan.
2. There is very limited research on biopolymers like chitin and chitosan.

3. SOLUTION METHODOLOGY

Present section tells about the details of research work to investigation of thermal properties of Narmada Riverside Crab shells based Chitosan. and obtainment of results.

3.1 Thermal Analysis of chitosan

Differential Scanning calorimetry (DSC) is a systematic procedure to decide the thermal properties of a chitosan (biopolymer). A DSC bend of chitosan was appeared in figure 4.1. the bend demonstrates that weight reduction happens in two phases. First stage begins around 85.44°C and the second stage begins around 309.60°C. the primary stage is appointed to the loss of water since polysaccharides more often than not have a solid proclivity for water and in this way might be effectively hydrated.

The second one compares to the warm decay of chitosn. the disintegration temperature of chitosan was observed to be 309.60°C. this outcome.

Demonstrates that chitosan exists as a steady structure toward thermal decomposition. In this investigation, the chitosan test was warmed at 25.00°C to 445.00°C interims of temperature. This is performed by instrument Perkin Elmer thermal investigation model CCLL-IDR-EQ-M-1031 Serial no. B550854318/B550854318. it was utilized under nitrogen gas air at stream rate of 10 ml/min. so as to limit thermo-

oxidative corruption. In all cases aluminum container were utilized to hold test with weight running from 6 to 7 mg.

Table 3.1 Parameters for DSC

S No.	Temperature pro-grammed	Values
1.	Introductory temperature °C	25.00
2.	last temperature °C	445.00
3.	Rate of warming °C/min.	10.00
4.	Cleanse gas	Nitrogen
5.	Stream rate of gas ml/ min	10.00
6.	Test measure, mg	5.209
7.	Test holder	Standard alumina 40

4. RESULT AND DISCUSSION

Present section contains the results and discussion about the research work. The details of the results obtained and discussion made based on of yielded results are displayed in these sections.

4.1 Results

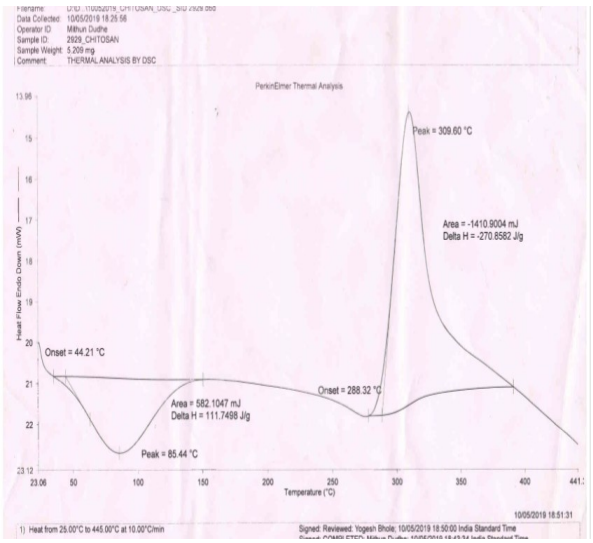

Figure 4.1 DSC analysis curve of Chitosan

A DSC bend of chitosan was appeared in figure 4.1. the bend demonstrates that weight reduction happens in two phases. First stage begins around 85.44°C and the second stage begins around 309.60°C. the main stage is allocated to the loss of water since polysaccharides more often than not have a solid partiality for water and along these lines might be effectively hydrated.

The second one compares to the thermal deterioration of chitosn.the decay temperature of chitosan was observed to be 309.60°C. this outcome shows that chitosan exists as a steady structure toward thermal degradation.

4.2 Discussion

Differential examining calorimetry (DSC) estimations were performed on a Parkin Elmer. DSC is a significant instrument for the assurance of glass change and liquefying conduct of chitosan (common biopolymer). Figure 5.1 demonstrates the DSC thermo grams of unadulterated chitosan. DSC thermo grams of chitosan test did not show any glass progress temperature which was principally connected with its inflexible crystalline nature and event of solid intra sub-atomic hydrogen holding. DSC thermo grams of chitosan showed an expansive exothermic with a pinnacle temperature at around 85.44° C, ascribable to the water holding limit, which in conceivable through the –OH and unsubstituted, free-NH_2 gatherings of chitosan. Also chitosan exhibites an expansive endothermic pinnacle temperature at around 309.60°C. As announced in the past investigation, thermal disintegration temperature of chitosan was higher than 250° C and it was viewed as that chitosan did not demonstrate a glass change before the deterioration temperature.

The improvement of thermally stable polymers is a region of broad continuous intrigue. With respect to numerous different materials, polymers have genuinely low use temperatures, which can decrease the utility of the item. This likely improvement in flame properties is, regularly balanced a lessening in procedure capacity and in ideal physical properties. Obviously, materials that are steady at high temperatures are probably going to be better exhibitions with respect to

as flame properties are concerned. The high temperature physical properties of polymers can be improved by expanding associations between polymer chains or by chain-hardening. Whatever the nitty gritty level of consistency of flame execution information from thermal decomposition date, its significance ought not be belittled; polymers can not consume on the off chance that they don't separate.

5. CONCLUSION, LIMITATIONS, AND FUTURE SCOPE OF THE RESEARCH

Present Section is contains the conclusion of research work, and limitations and future scope of the research work, details of which are presented in upcoming sub-sections.

5.1 Conclusion

This examination have effectively shown to examination the thermal properties of Narmada riverside crab shell chitosan by utilizing Differential Scanning Calorimetry Technique, chitosan effectively arranged from chitin. Biopolymers (chitosan) regularly show expansive dissolving endotherms and glass changes as major scientific highlights related with their properties both the glass and liquefying advances are unequivocally wards on handling conditions and scattering in auxiliary and synthetic properties of chitosan. Portrayal of chitosan requires a nitty gritty investigation of these trademark thermal advances utilizing differential scanning calorimeter (DSC).

1. First Stage Degradation begins around 85.44°C and Delta H = 111.7498 J/g
2. Second Stage Degradation begins around 309.60°C. And Delta H = -270.8582 J/g

5.2 Limitations and Future Scope of the Research

Following are the limitations of the present research work:

1. The research work is limited to a specific Narmda Riverside crab because crab are less in quantity.
2. Extraction of chitin requires chemical engineering knowledge.
3. Low mechanical strength of biopolymer.
4. Chitosan decomposed before melting.
5. Difficult to achieve chitin deacetilation.
6. Practical difficulties in DSC.

Following points represent the future scope of the research work:

1. The different properties of chitosan, for example, dampness assimilation, weakness and tribological conduct might be resolved utilizing broad experimentation.
2. The analyses can be reached out by organic extraction of chitin and chitosan.
3. The examination can be stretched out by other warm investigation techaniqes like that TGA, TMA, DMA and so on.
4. The analyses can be reached out by Fourier change infrared spectroscopy (FTIR).
5. The analyses can be reached out by estimations of water vapor transmission rate.
6. The analyses can be reached out by nuclear power microscopy.

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