



Investigation of Partially Methyloated Polyacrylamide Guar Gum by FTIR and Thermal Properties

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Author's contribution

The sole author designed, analyzed and interpreted and prepared the manuscript.

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ABSTRACT

Cotton samples were modified with the reactive composite with different methylation percentage varying from 20 to 80%. The Fourier transforms infrared (FTIR) spectra of modified cotton fabrics have followed at each condition which had been prepared via graft copolymerization of acrylamide and guar gum using the KBrO₃/Thiourea redox system in the presence of ammonium chloride as catalyst. The thermal properties of the samples were analyzed using Thermo Gravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) techniques. TGA tested results indicates that the thermal stability of modified cotton fabric with 40% methylation got enhanced than the blank, the relative TGA results show that the char residue of cotton sample after 400°C is higher than the blank one which means lesser volatile hazardous components. DSC measurement shows variation in the thermal behavior according to different methylation percentage.

Keywords: Acrylamide; TGA; FTIR; guar gum; DSC; methylation and cotton fabric.

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1. INTRODUCTION

The most used material for textile manufactures are cotton (cellulose based material). It is used in furniture's, packing paper and medical products [1] by converting it to cellulose derivatives and regenerate materials, because of cellulosic materials good mechanical properties; low cost, light weight, moderate strength, high specific modulus, renewability, biodegradability, lack of health hazards and recycle ability [2]. Textile materials and products are suitable for many medical and surgical application in which a combination of strength, flexibility and sometimes moisture and air permeability is required. Materials used include monofilament and multifilament yarns, knitted, woven and non-woven fabrics and composite.

The main reactions acting on the cellulose structure and causing its alteration are photo-degradation, acid hydrolysis, oxidation and biodegradation. A complete analysis of them is quite complex since these phenomena are all related to each other [3]. Graft copolymerization of vinyl monomers on to cellulose has been widely investigated [4,5] in particular the acrylic monomers can be used because of their physical characteristics (water –repellence transparency, good flammability) the reaction of free radicals in the cellulose chain is necessary to imitate the grafting, the polymerization can be induced by chemical method, more contently by reaction as X-ray, Uv-light, γ -ray. Electron beam the selection of a particular type of radiation on the degree of pentation required. Since natural fibers from plants such as cotton when heating decompose and totally degrade losing the main feature improving thermal stability is highly advantageous.

Natural poly saccharide has recently been suggested that biodegradable plant polysaccharides such as pectin dextran and galctomannan (by forming (1-4) cross linkage hydrogels) to reduce water solubility. Guar gum (GG) is a natural polysaccharide made of linear β -(1-4)-mannose backbone to which α (1-6) linked galactose residues are attached as a single unit side chains [6] and glycosidic linkages, in the ratio 2:1. Chemically guar gum (GG) backbone has three reactive OH groups present in each anhydrous glucose units which can be replaced by viscous desirable functional group with different degree of substitution can be obtained in a sugar moiety [7-9]. It is the most commonly used natural gum because of its cast

effectiveness and thickening effect. Due to its low cost, nontoxic, biodegradability, biocompatibility, high viscosity and water-solubility, GG is now used in many industries [10]. As sizing; finishing agents in the textile and paper industries as a binder stabilizer and thickener in the cosmetics; food industries and as a fracturing fluid additive in mining and hydraulic fracturing processes [11]. However, substitution of GG is difficult task due to insolubility to offered dispersion without degradation so in that work try to modify of GG with methylation and investigated with thermal properties by using ammonium chloride as catalyst with time of curing 160°C for 5 minutes onto cotton fabric [12].

In particular the acrylic monomers can be used because of their physical characteristics (water – repellence, transparency, good flammability. The cretin of free radicals in the cellulose chain is necessary to initiate the grafting. The polymerization can be induced by chemical method or by radiation (as UV, X-ray). In our research acrylamide (Aam) based polymers are important class of water soluble polymers which have a very broad range of industrial applications. Polyacrylamide (PAam) is widely used in flocculants, adhesives, viscosity control agents for oil recovery, latex, paint systems, pigment printed from textiles and coating. Stiffening fabrics can be produced by coating it with a hot adhesives such as polyester, polyamide, polyolefin, natural or synthetic wax prefer by containing an epoxy resin [13]. Stiffness can be induced through very interesting and easy processes which involve impregnating of fabric in a starch / formaldehyde adducts in the presence of catalyst [14]. The main advantage of the inverse emulsion polymerization in comparison to bulk or other polymerization techniques is the fact that this process enables one to reach both high molecular weights and high rates during polymerization [15]. Although inverse emulsion polymerization has been recognized as being very useful in terms of technological applications and scientific investigation up to now studies on this topic have mainly focused on the macroscopic kinetics mechanisms [16-19] which interactions on molecular level have occurred between the monomers and the inverse micelles and how these interactions affect the polymerization process have been great importance for a deeper understanding of the polymerization in inverse emulsion [20,21,22].

Thermal analysis techniques are often used for characterization of polymeric materials, especially in the range of 100°C and above, differential scanning calorimetry (DSC) allows a rapid detection and measurement of the physico-chemical changes that a polymer undergoes when subjected to heating whereas, thermogravimetric (TGA) analysis provides a method for determination of mass change in the polymer as a function of time and temperature [23]. Thus, these techniques reflect reactions which occur at the molecular level of the materials. In recent years, copolymers and blends attracted the attention of material researchers to obtain intermediate specific functions. These interesting properties are attributable to the molecular motions allowed in their amorphous phases. The interphase regions in blends and copolymers are very important. Depending on the chemical nature of the doping substances and the type and the extent in which they interact with host matrix, the doping alters the physical properties to different degrees [24]. Science natural fibers from plants, such as cotton and flax when heated, decompose and totally degree losing the main features, improving their thermal stability is highly advantages [23].

Fourier transform infrared (FTIR) spectroscopy has been used for decades as a powerful tool for the identification of organic as well as inorganic material in art technology commercials FTIR instruments offer a big variety of methods for different analytical questions in order to generate usable spectra [25]. The diamond cell of FTIR microscope as well as FTIR-ART spectroscopy have been applied frequently for the analysis of paintings both requiring only minute amounts of sample material in many cases sampling is possible especially from paintings subjected to a resolution treatment however, nondestructive method would be preferable in recent years reflection FTIR instruments for non-destructive in situ analysis of art objects have been developed and applied controlling the optical path either by mirrors [26] or fiber optics [27].

In recent years, copolymers and blends have attracted the attention of materials with respect to homopolymers for some specific functions. These interesting properties are attributable to the molecular motions allowed in their amorphous phases. The interphase regions in blends and copolymers are very important. Depending on the chemical nature of the doping substances, the type and the extent in which they interact with host matrix, the dopant alters the physical properties to different degrees. In

our research guar gum has grafted by acrylamide in the presence of redox system from bromide bromate mixture to reproduce natural reducing agents and evaluate the thermal and FTIR of modified cotton with that produced polymer.

2. EXPERIMENTAL

2.1 Materials and Chemical Used

Mill scoured and bleached plain weave cotton fabric (100%) weight 120 gm/m², thickness was 0.2 mm, number of yarn/cm in warp direction 30 and in weft 31. were supplied by El-Naser company for spinning weaving and dyeing. Mahalla El-Kubra Egypt.

Guar gum (GG) was purchased from S.D. fine chemicals (Mumbai, India) and was used without further purification. Acrylamide, potassium bromate (KBrO₃), thiourea (TU), sulfuric acid, sodium hydroxide (NaOH), triethanolamine (TEM) and formaldehyde (HCHO), acetic acid (CH₃COOH) and ammonium chloride (NH₄Cl), potassium persulphate were laboratory grade chemicals.

2.2 Preparation Methods

2.2.1 Preparation of polyacrylamide guar gum (PAam-GG) composite

The composite was prepared by dissolving GG in acidic water (using sulphuric acid 2N) at pH 3, with stirring for 10 minutes then add calculated amounts of KBrO₃/TU (6/6 m mol/ 100 gm GG) with gradually increase temperature up to 50°C then adding monomer (Aam) (30% based on weight for 2 hours with liquor ratio 1:30. After that Re-polymer further treatment by adding suitable amount of ammonium persulphate to reach the suitable viscosity to be used onto fabric at 70°C for 3 hours with stirring and reach complete conversion of monomers.

2.2.2 Preparation of partially (N-methylated of polyacrylamide guar gum (PAam-GG) composite

The result composite formed neutralized with sodium hydroxide and adjusted pH at 9.5 by using triethanolamine (TEA), then heated to 50°C with stirring for 20 minutes then add calculated amount from solution (HCHO at pH 9.5) to poly(Aam-GG) composite then continues stirring for 3 hours at 50°C., to obtain different methylation percentage (20,40,60, 80%) based on weight of Poly(Aam-GG) composite was prepared.

2.2.3 Fabric treatment

Cotton fabric samples were immersed in reactive composite prepared with different methylation percentage based on weight of (N-methylol - PAam-GG) content in the presence of catalyst 0.5% ammonium chloride as catalyst then the samples padded to wet pick up 100% then dried at 80°C and cured temperature 160°C for 5 minutes.

2.3 Fourier Transform Infrared (FTIR) Spectroscopy

The FTIR spectrum of the pigment printing fabrics are recorded using Nicolet 380 spectrophotometer with Attenuation Total Reflection (ATR) mode with a zinc selenide crystal, in spectral range 4000-650 cm^{-1} , to insure reproducible contact between the crystal face and the fabric pressure about 18 Kpa [28] was applied to the crystal holder, with resolution of 4 cm^{-1} and a number of scans of 32 were acquired for each spectrum. Minute samples were collected at brittle areas of the paint-layer using a syringe needle (0.3 mm diameter) and measured on diamond cell (spectra tech. Shelton, Ct. USA). The obtained transmission spectra and will be termed as μ -FTIR spectra in the following parts of the publication.

2.4 Thermal Gravimetric Analysis

The thermal behaviors of pure cotton and the reactive composite (methylated PAam-GG) samples were investigated with Shimadzu TA-60 (Kyoto, Japan) for thermogravimetric analysis (TGA). The thermogravimetric curves were differentiated with respect to time concurrently with an analogue- derivative unit. Shimadzu DSC-60 analyzer (Kyoto, Japan) was used for Differential Scanning Colorimetry (DSC), which were carried out on all samples at a heating rate of 10°C/min. with dry nitrogen (N_2) as the carrier gas, and at a flow rate of 20 ml/min. Sample runs were repeated at least three times to ensure the reproducibility scans were started at temperature at 10°C and run to a final temperature 650°C.

3. RESULTS AND DISCUSSION

3.1 FTIR-ART Spectral Analyses

FT-IR spectroscopy has long been recognized as a powerful tool for the structural information. The

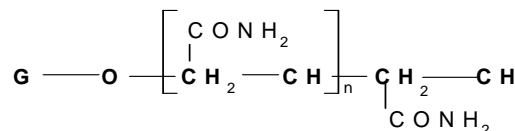
position, intensity and shape of vibrational bands are useful in clarifying conformational and environmental changes of polymers at the molecular level [29]. FT-IR spectra of methylated PAam-GG composite and PAam-GG composite were presented and the assignment of the main vibrational modes was listed in Table 1.

A few new characteristic peaks were obtained the stretching and bending vibrations. It is clear that, the characteristic absorption band of blank cotton at wave length $\sim 1465 \text{ cm}^{-1}$, $\sim 1715 \text{ cm}^{-1}$, $\sim 2986 \text{ cm}^{-1}$ and $\sim 3475 \text{ cm}^{-1}$, represent the signals of the following functional group CH bending, C=O antisymmetric, CH vibrating and OH stretch groups, which represents in (Fig. 1), the most characteristic functional chemical groups present of cotton fabrics samples under investigation were analyzed for their chemicals groups [30,31,32].

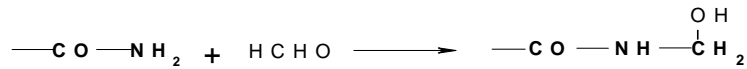
The peak intensity values of the major functional groups exist in blank cotton samples; (Transfer FTIR=NRT spectroscopy): CH bend at 1340 cm^{-1} , C=O antisymmetric at 1600 cm^{-1} , CH vibrating at $\sim 2900 \text{ cm}^{-1}$, and OH at $\sim 3300 \text{ cm}^{-1}$, these bands having the following intensity 4.4, 1.4, 1.1 and 5.6 respectively as in (Fig. 1).

The FTIR spectrum for native guar gum in (Fig. 2) showed broad band in region of 3431 cm^{-1} were attributed to stretching vibrational of large number of free hydroxyl groups in the guar backbone. And also, the major absorption bands together with its intensity are CH bending $\sim 1340 \text{ cm}^{-1}$ having intensity 3.22, C=O antisymmetric at $\sim 1600 \text{ cm}^{-1}$ intensity are 3.04, CH stretching at $\sim 2900 \text{ cm}^{-1}$ having intensity 2.99, and OH stretching at $\sim 3300 \text{ cm}^{-1}$ with intensity 4.25 as appeared in (Fig. 2).

Polymerization of guar gum with acrylamide to produce polyacrylamide guar gum (PAGG) as in shown below.



Methylation process was carried out to change the functional group (- CONH₂) to form CH₂OH according to the following equation.



to activate the produced function groups via NH_4Cl as a catalyst to infant the purpose of increases the bonding on cotton fabric to improve its characteristics to be flame retardant.

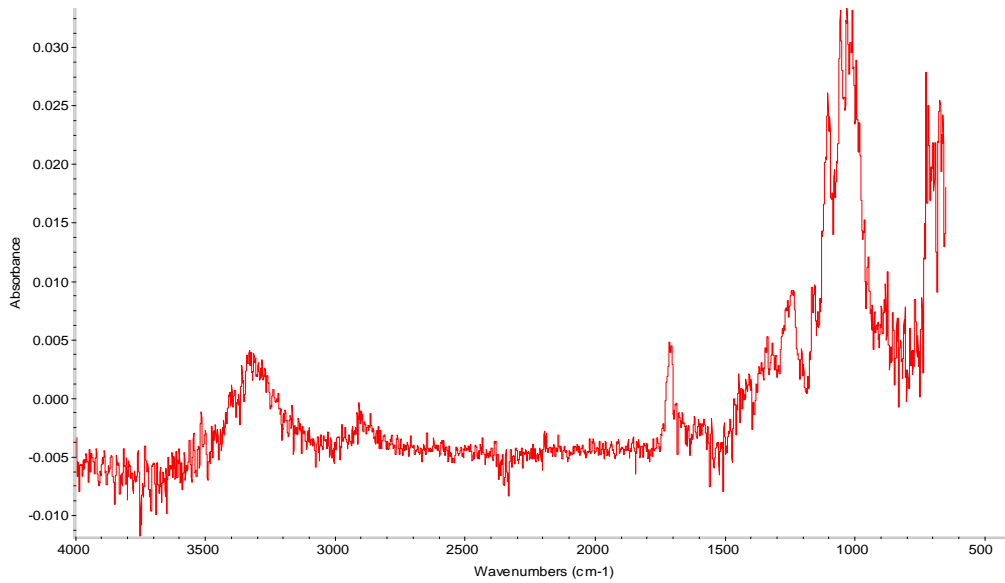
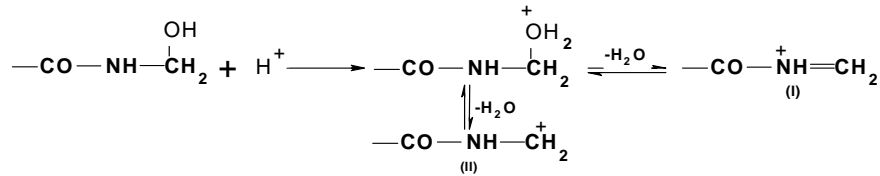


Fig. 1. FTIR –ATR spectra diagram of blank cotton

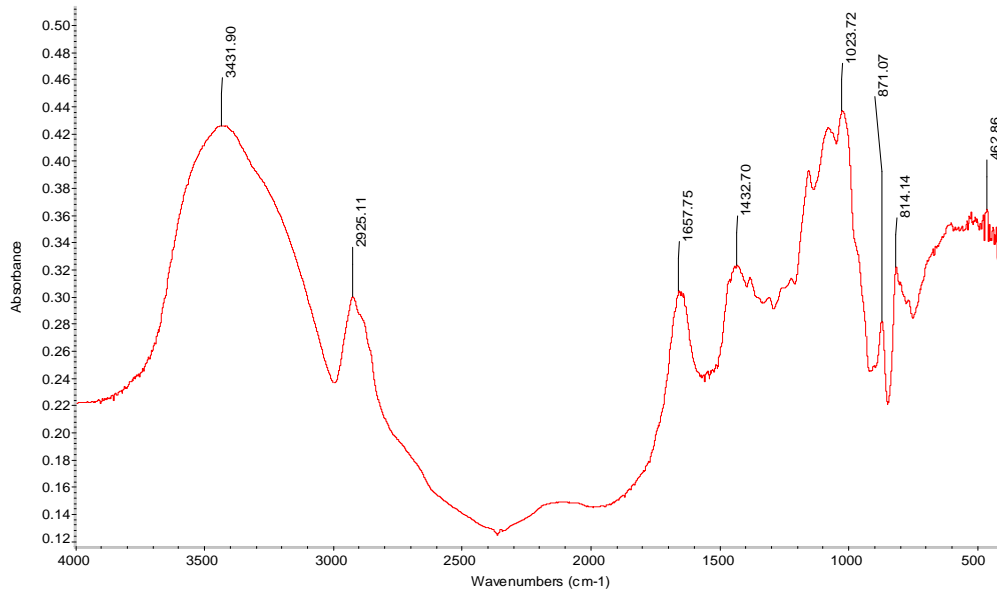


Fig. 2. FTIR –ATR spectra diagram of native guar gum (GG)

Different concentration of formaldehyde were used ranging from 20: 80% to determine the best concentration for such treatment as appear in (Fig. 3) showed that FTIR spectroscopy of cotton samples after treatment with different concentration of formaldehyde hydration new. The following changes were appeared NH amide appeared at ~3400 and overlapping with OH stretching that appeared on cotton before treatment, after 40% formaldehyde disappeared overlapping due to the decrease in NH and group.

The results of the table and reaction that of the best condition for the reaction to pass reversibly and the chance for the increasing the production of these composite compounds (I) or (II) adhere

lightly on fabric. Cotton fabric OH group is nearly ~40% CH₂O whereas this ratio highly enough to combine with H⁺ in NH₄Cl; beside these results are indicated for the peak intensity values of the functional groups CN (amide III), NH bend (amide II) at $\gamma \approx 1586$ and 1020 respectively.

The presence of bands in case of reactive PAam-GG polyblends compared to that of GG and cotton fabric conforms the successful grafting of PAam-GG chain onto the backbone of GG these important peaks elucidating the structure of GG and methylolated PAam-GG (reactive composite). These important peaks elucidating the structural have been summarized in Table 1.

Table 1. Comparison FTIR bonds of cotton, guar gum and reactive composite (PAam-GG)

Reactive composite	Wavenumber (cm ⁻¹)		Assignment group
	cotton	guar gum	
1325.73	1317.44	1023.72	Associated with C-O-C stretching group
1665.67	1715.31	1657.75	C=O stretching vibration of aliphatic esters
2998.34	2986.29	2925.11	Symmetrical stretching modes of C-H bonding
3509.76	3475.26	3431.90	Hydrogen bonded and hydroxyl O-H group
Not found	1465.74	1432.70	CH (bending)
3435.00	Not found	Not found	NH (stretch)
1642.96	Not found	Not found	NH (amide II)
1245.33	Not found	Not found	CN
3607.00	Not found	Not found	OH (free)

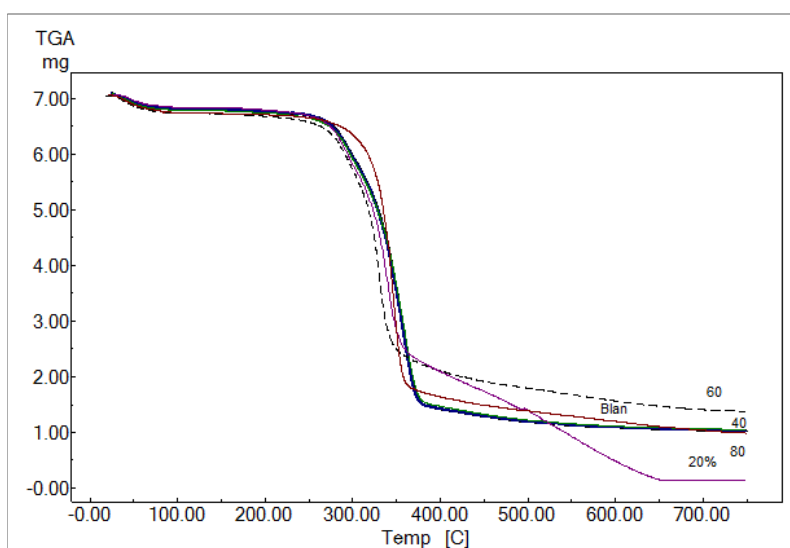


Fig. 3. Thermo-gravimetric analysis (TGA): Cotton and modified cotton with reactive composite samples under different methylation parentages (20, 40, 60 and 80% HCHO with catalyst ammonium chloride)

3.2 Thermal Analysis

3.2.1 Thermogravimetric analysis

Thermogravimetric (TGA) is widely used to investigate the thermal decomposition of polymers to determine the thermal decomposition kinetic parameters such as activation energy and reaction order. These parameters can be used to obtain a better understanding of thermal stability of polymers. Also, TGA enables to determine values of the moisture loss of fibers in addition to that obtained by conventional method. The recorded (TGA) plots all samples coexistence of more than one degradation process.

Guar gum, chemically known as guar galactomannan [33], consists of a linear chain of (1→4)-linked α -D-mannopyranosyl sugar units with (1→6)-linked β -D-galactopyranosyl sugars as single unit side chains [34]. Studying the thermal properties of guar gum is important to determine its thermal stability and to distinct weight loss regions similar to previous thermal studies of guar gum [35]. The first weight loss region is located between room temperature and 150°C. It is attributed to the evaporation of adsorbed water, which accounts approximately 11–13% of the sample weight, and lies within the range reported previously [36]. The second weight loss region, located between 200°C and 400°C, is attributed to the degradation of the galactomannan chains. The percent weight loss in the second weight loss region is in the range of 59–77% of the sample weight. The lower temperature loss (first step) may have corresponded to the breaking (1-4) linkage and the second to the third step may have corresponded to the degradation of the whole polymer.

Then the thermal degradation rate reduced with increase in temperature, while the char residue got decreased slowly. This phenomenon resulted

from further thermal degradation of formed char. The cotton fibers coated by reactive composite in the presence of catalyst ammonium chloride with different percentage of formaldehyde showed a lower start degradation temperature, compared with untreated cotton fabric. The improved thermal stability was assigned to the presence of the methyolated reactive composite coating, which protected the underlying cotton from degradation. The fabrics treated for 20,40,60 and 80% showed a homologous trend with the enhanced the start degradation temperature (334°C).

The kinetics parameters for uncoated and coated cotton fabric obtained from this (Fig. 1) and listed in Table 2. Thermal gravimetric analysis of the cotton fabric (blank) indicates that the start degradation temperature was about 200°C and its main mass loss stage was at the temperature range from 200 to 749°C resulting from the polymerization of cellulose through transglycoation. The weight loss in the first step corresponds to the evaporation of bound water (the desorption of H₂O physically bond to fiber and dehydration of cotton) this process results from it a sharp endothermic peak and the initial weight loss starting from room temperature up to 749°C it will know that the melting temperature of cotton decrease with increasing moisture content [37].

The second step starting above 200°C, the composite fixation on the cotton fabric may be associated with decomposition rate of aqueous ammonium chloride catalyst with the formation of more hydrogen proton and rapid evaporation of NH₃ molecules during the curing would accelerate the cross linking reaction of the reactive composite with cellulose hydroxyls, in thermal reaction, molecules in their ground state can be raised to higher vibrational levels of electronic ground state by collision with other molecules or wall the thermal energy is factor

Table 2. The weight loss of the blank cotton (unmodified) and modified cotton with different concentration of HCHO %

Sample	First step		Second step		Third step	
	weight loss %	Mid point (°C)	weight loss %	Mid point (°C)	weight loss %	Mid point (°C)
Blank	29.363	334.94	50.873	361.68	6.011	378.67
20%	22.139	292.77	27.088	345.91	7.446	467.53
40%	20.946	278.68	20.359	298.24	61.415	349.47
60%	15.015	276.77	16.224	287.95	60.324	345.74
80%	10.503	269.68	15.239	287.85	45.546	335.2

controlling the frequency and probability of such collision. Chemical change occurs when a particular bond accumulate sufficient energy for bond dissociation. In general the degradation of polymer s is a heterogeneous reaction on which the increase the temperature causes loss of mass.

Form Table 1 representing that the TGA of all samples, it noticed that the first step of weight loss increase for samples of cotton which are modified with reactive methylolated (reactive composite) from 20% to 40% then decrease from 60 to 80% percentage of HCHO above the blank sample. The second step of weight loss (decomposition state) the weight loss occurs for the blank cotton sample (50.873%) of char residues and the weight loss increased from 20% to 40% HCHO with using NH_4Cl then decreased again from 60% to 80% with the lowest value (15.239%) of char residues this means that, the best percentage of modification with 40% HCHO. The third stage of decomposition showed that the lowest value of the weight loss in this stage is that for blank cotton sample (6.011%) and the value of weight loss also increase up to 40% HCHO then decreased again, it is clear that the difference between the weight loss at this stage compared to the blank is due to the percentage of formaldehyde percentage used.

Then the thermal degradation rate reduced with the increasing of temperature, while the char residual amount decreased slowly. This phenomenon resulted from further thermal degradation of formed char. The cotton fibers coated by reactive composite with different percentage of formaldehyde 40% showed a higher onset degradation temperature, the improved thermal stability was assigned to the presence of the inorganic–organic hybrid coating [38], which protected the underlying cotton from degradation. The fabrics treated for 20, 60 and 80 % HCHO showed a homologous trend with

the enhanced the onset degradation temperature. However, the cotton fabrics treated with 40% HCHO showed a lower relative to that of the uncoated cotton fabrics from Table 1.

The value of total weight loss at the end of thermal degradation of the samples under test by char residue as follow (char residue = $W_1 - W_2$) where W_1 is the total weight of the sample at the beginning of the thermal process and W_2 is the total weight loss of the sample. If the weight loss of the char residue is increased, it behaviors as a carbonized replica of the original fabric and function as thermal barrier, the decomposition temperature of the fabrics is enhanced and the formation of flammable volatiles is decreased and the fabrics had a good flame retardant when exposed to fire.

3.2.2 Differential Scanning Calorimetry (DSC)

The differences in DSC parameters can be related to the matrix material, i.e. the non-helical parts of the intermediate filaments, the material between the filaments and all other amorphous, microbiological components. DSC had been used to measure temperature and heat of transitions specific heat (which is a paramount thermal property), thermal-emissivity and certain isothermal functions [39], with these general headings fall the measurement of glass transition (T_g) crystallinity including the measurement of enthalpy of melting, purity, rate of reactions, rate of crystallization and rate of decomposition. The first observed of ($T_g = 54.36$) followed by appearing big peak corresponding to water evaporation. This peak appears at around 90-100°C which is associated with the denaturation of the helicoidally material appears at around 334°C. Peaks are characterized by the peak temperature and the area of the peak. The glass transition is the second order transition caused by relaxation of the chain segments in the amorphous portion of the polymer [40].

Table 3. The weight loss of the blank cotton (unmodified) and modified cotton with different concentration of methylolation %

Sample	First step		Second step		Third step	
	End set Temp. (°C)	Heat of fusion Heat release (J/g)	End set Temp. (°C)	Heat of fusion Heat release (J/g)	End set Temp. (°C)	Heat of fusion Heat release (J/g)
Blank	191.00	-65.00	-----	-----	-----	-----
20%	90.00	-9.74	338.88	-336.06	365.61	7.67
40%	83.53	-29.15	353.3	-809.49	364.91	42.84
60%	76.87	-8.23	356.59	-251.00	346.03	-7.77
80%	87.36	-8.00	345.93	-172.41	362.92	1.83

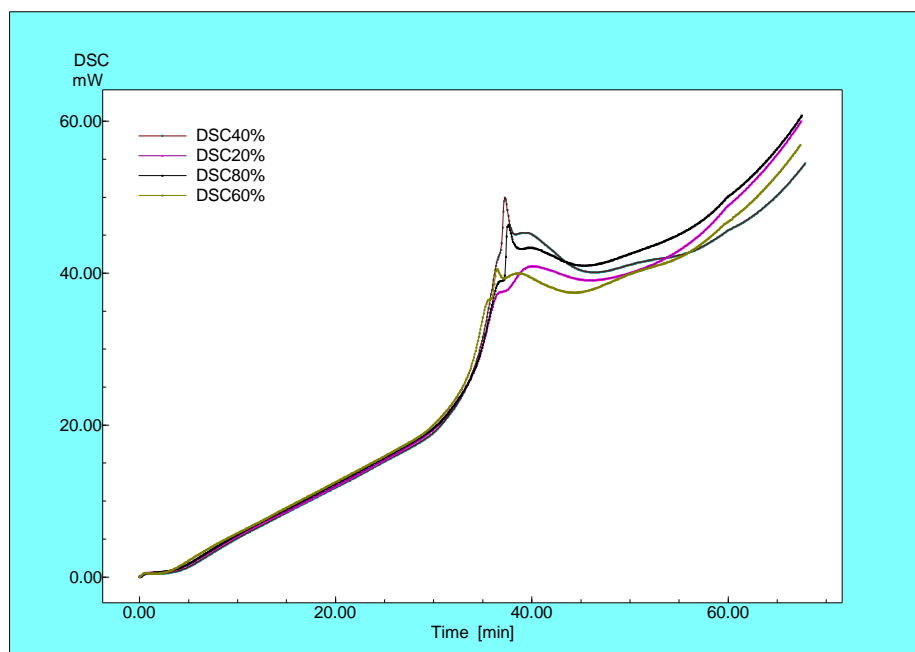


Fig. 4. Differential scanning calorimeter (DSC) of cotton and modified cotton with reactive composite samples under different methylation parentages (20, 40, 60 and 80% HCHO)

The DSC curve exhibits a significant start from 200°C which is corresponding with TGA Fig. 2 however, the endothermic starting for 200°C with its peak around 334.94°C.

This results from melting of anhydroglucose rings of the cellulose molecule. The end set temperature of blank cotton fabric is the highest (190°C) while decreased the end set temperature for modified cotton fabric around (90, 83.53, 76.87 and 75.36°C) respectively from 20 to 80% HCHO used. The reduction of the modified cotton flammability was attributed to the cross link covalent bonding of the modified to the anhydroglucose rings of the cellulose molecule thus interfering with the pyrolysis of cellulose to levoglucosan.

From Table 3 shows the values of heat of fusion (heat release) J/g which is represented by the area under the endothermic peak of the decomposition step, and its rate has been found to be very effective to evaluate fire hazards. it can be detected that for the first step. The heat release value of the cotton fabric coated with reactive composite with 20% methylation is (-9.74 J/g) while that with 40% methylation heat release is (-29.15J/g) then the heat release value increase with 60% and 80% methylation percentage to (-8.23 J/g) and (-8.00 J/g)

respectively. For the second step (decomposition step) the highest value of heat release (-809.49J/g) for the 40% methylation percentage followed by 60% and 80% methylation percentage with the value (-251.00J/g and -172.41J/g) respectively. The decomposition temperature of any polymer depends on its molecular weight and its purity; it also affected by its morphology. This decomposition temperature depends on the crystallinity of the polymer, the higher crystallinity has the higher decomposition temperature.

4. CONCLUSION

In this study, investigated the groups formed using FTIR analysis during modified with reactive composite (PAam-GG) with catalyst NH_4Cl with curing temperature 160°C and curing time 5 min. And also, studying thermal analysis technique which is very accurate tool to difference between the thermal stability of cotton fabric samples after modified with reactive composite and different methylation percentage from (20% - 80%). DSC and TGA curves showed that the mechanism of these methylation percentage converts the cotton fabric to carbonaceous residue or char when exposed to high temperature and hence reduce the volatile formation. The initial decomposition

for all the samples is nearly comparable, while the heat release (J/g) differ from one sample to another and the highest value is for 40% methylation.

COMPETING INTERESTS

Author has declared that no competing interests exist.

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