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Thermogravimetric and Fourier transform-infrared analysis of some selected plants' mucilage for pharmaceutical applications

Taleat Tella A.A[1]*, Lajide L[2] and Owolabi B. J[3].
[1]Science Laboratory Technology Department, Federal Polytechnic, Ede.
[2]Department of Chemistry, Federal University of Technology, Akure.
tellawale@gmail.com

ABSRTACT - ***Mucilaginous powders were obtained from different plants: Cissus populnea stem, wild mango seeds (Irvingia gabonensis) after lipid extraction and fruit pulp of Baobab (Adansonia digitata) tree. The different mucilage powders obtained after purification was subjected to thermal analysis using Thermal analyzer (Pyris TGA Perkin Elmer, U.S.A), Fourier transform-infrared analysis was done with spectrometer (Perkin-Elmer) and elemental analysis with (PE 2400 Series) elemental analyzer. Two steps of pronounced weight loss were observed during thermal analysis of all samples. The first differential thermograph peaks of all samples were above 100^0C and the first weight loss may be attributed to loss of moisture from the samples with weight loss of between 8 and 10%. The second weight loss was found to be above 50% with differential themogravimetric peaks above 200^0C indicating that the mucilage powder samples were stable at high temperature. The initial decomposition temperatures determined from the thermographs were 250^0C, 260^0C and 260^0C respectively for Cissus Populnea, Irvingia gabonensis and Adansonia digitata powders. This implies that the powders can withstand industrial processes at high temperatures. The kinetic parameters of degradation as obtained by the application of Arrhenius equation gave the activation energies of between 8.86 to 29.12 kJ/mol while the frequency factor was found to be from 0.82/sec. to 1.49/sec. The elemental analysis indicated the presence of certain percentages of carbon, nitrogen, sulphur and hydrogen in all the extracted mucilage powders. Fourier transform-infrared analysis gave peaks that indicated the presence of N-H, C=O, and O-H functional groups which are typical of polysaccharides.***

Keywords: Mucilage, Thermal analysis, FT-IR analysis, Arrhenius plots, kinetic parameters.

1.0 Introduction

Polysaccharides are widely used in different pharmaceutical and food formulations either as binders, suspending agents, thickening agents, emulsifiers and gelling agents because of their renewability, availability, non-toxicity, biodegradability and compatibility with most drug actives and food substances. Polysaccharide mucilage and gums used in pharmaceutical and food industries include guar gums, tagacanth, acacia gum (gum Arabic) and grewia gum amongst others. Some selected plants parts have been used as soup thickener in some African countries and indeed in Nigeria mainly because of their swelling ability in solution as well as their non-toxicity. These properties of some plants parts (leaves, stem or seeds) have endeared their usage in the preparation of some African local delicacies.

Studies have been conducted on the nutritive values of some of the plants with mucilaginous properties. Dietary value of okra mucilaginous was reported (Ihekoronge and Ngoddy, 1985). Medicinal benefits of Wild Mango (*Irvingia gabonensis*) seed extract on bacteria and fungi was also reported (Kuete *et al*, 2007) while the mineral compositions of some vegetables have been investigated (Rice-Evans and Miller, 1985, Steyn *et al*, 2001 and Oulai *et al*, 2014). The industrial applications of most mucilaginous plants were examined for their physico-chemical properties and were found to be quite useful (Ofoefule, 2001). The use of okra mucilage in sustaining release of drug was reported (Zaharaddin *et al*, 2014). Studies on the mucilage extracted from okra fruits and baobab leave was reported (Woolfe *et al*, 1997).

The dietary potential of *Corchorus oliturus* and okra have been studied (Maramang, 2013). However, good as the reported potentials of plant mucilage in the industrial processes were, there are paucity of information on the thermo-gravimetric analysis of some of these plants gums and mucilage. Although some of the plants from which these priced natural polysaccharides are extracted can be identified by their morphological characteristics, it is not always possible to identify the dried mucilage powder from a particular plant source therefore. a simple and rapid method for the determination of Fourier transform-infrared (FT-IR) and thermal stability properties of some mucilage powders are highly required. The present study therefore seek to obtain information of the FT-IR characteristics and the thermal stability of mucilage powder extracted from *Cissus populnea* stem, *Irvingia gabonensis* (Wild-mango) seed and *Adansonia digitata* (Baobab tree) fruit pulp as a guide to their application in industrial processes.

Fourier transformation infrared spectroscopy is a non-destructive analytical technique that allows the reliable and fast determination of several properties of samples without sample pretreatment. The FI-TR spectra of organic compound provide a unique *fingerprint* which can easily be differentiated from the FT-IR pattern of other compounds (Yiling, 2012). The characteristics absorption bands can be used for the specific detection of some functional groups in the compound as reported by (Menezel and Prime, 2009, Roy *et al*, 2009 and Mehrotra *et al*, 2007).

Thermo-gravimetric Analysis (TGA) also has advantage of reliability, reproducibity and rapidity of results generation over broad temperature range and heat rates. This method has been used for chemical composition analysis and structures of sample (Menezel and Prime, 2009). The method has been applied to the thermal analysis of energy compounds in plant like lipid and polysaccharides (Zhang *et al*, 2009, Manrel *et al*, 2009). Thus, the FI-TR and TGA can provide substantial information on the identity and thermal stability of mucilage powders for various applications.

2.0 Materials and Methods

2.1 Materials

Mucilage were extracted from stem of *Cissus populnea* plant and defatted *Irvingia gabonensis* (wild mango) seed by soaking the respected plant parts in distilled water after thorough washing. The obtained viscous solution in each sample was passed through muslin cloth and the mucilage was precipitated out by addition of 95% ethanol in the ratio 1:3 by continuous stirring. The coagulated mucilage formed as a whitish mass was transferred to an evaporating dish, treated successively with ethanol, dried in an oven at temperature of 45^0C, powdered and stored in airtight containers with appropriate labeling. The obtained powdered mucilage samples were labeled thus as: *Cissus* mucilage powder (CMP) and Wild-mango mucilage powder (WMP). The *Adansonia* fruit pulp powder was obtained by scraping the pulp from the ripped, dried fruit which has been broken by sledge hammer. The obtained pulp was screened through muslin cloth to removed extraneous materials and labeled as *Adansonia* fruit pulp powder (APP).

2.2 FT-IR Spectroscopy

The structural characteristics of the extracted samples were determined by FT-IR Spectrophotometer (Perkin-Elmer Corp. U.S.A). 10 mg of sample was milled in a mortal with 100 mg of dried spectroscopic potassium bromide (KBr) powder and then pressed into pellets in preparing a salt disc (10 mm/dm) for transforming IR spectral measurement in the frequency range of 4000-500 cm^{-1}. The reference spectra were obtained from the cleaned blank crystal prior to the analysis of each sample replicate and samples were run in triplicate within a single day.

2.3 Elemental analysis

The elemental analysis on carbon, hydrogen, nitrogen, and sulphur weight percentages of the extracted mucilage powder was performed with (PE 2400 Series) Analyzer. 1 mg of sample was injected and eluted with Helium and detected by Thermal Conductivity Detector.

2.4 Thermo gravimetric analysis (TGA)

Thermogravimetric analysis was performed by thermogravimetric analyzer. About 10 mg sample was placed in silica pans and the samples heated at 10^0C/Min. from the ambient temperature to 600^0C. The analysis was carried out in lean air (nitrogen) environment. The presence of oxygen encourages the thermal decomposition of samples at lower temperatures (Fancioso *et al*, 2005). The results of TGA analysis were expressed on a dry weight basis and the TGA curve and derivative curve of each run was automatically corrected with the baseline of a blank experiment (Brown, 2001). TGA is a simple and accurate method for studying the decomposition pattern and thermal stability of polymers. The details of thermal properties according to the primary thermographs and derivative thermographs are given in table 2.

3.0 Results and Discussions

3.1 Tables of Results

Table 1: General Specifications of Natural Mucilage Used

Parameters	CMP	WMP	APP
Appearance	Dark Brown	Grey	Cream
Solubility	Warm H_2O	Warm H_2O	Warm H_2O
Swelling Index (%)	140±12.23	120±15.23	135±17.35
Viscosity (1%w/v(Cs)	17.60±2.24	15±3.12	13±3.45
Microbial Count (10^{-6}) folds	90±15.34	200±20.14	205±22.12
pH	6.35±0.50	6.25±0.80	5.25±0.45
Loss on Drying (%)	7.33±1.20	5.45±1.55	6.63±1.45
Bulk Density	0.62±0.15	0.84±0.20	0.53±0.13
Tapped density	0.95±0.20	1.04±1.10	0.76±0.25
Hausner Ratio	1.53±0.06	1.24±0.45	1.430.53
Angle of Repose (degree)	24±3.13	29±5.15	23±4.17
Total Ash (%)	6.30±1.15	4.30±1.54	3.551.23

a b c

Plates 1: (a) Defatted *Irvingia gabonensis* seed mucilage powder (b) *Adansonia. digitata* Fruit pulp powder (c) *Cissus. Populnea* (stem) mucilage powder

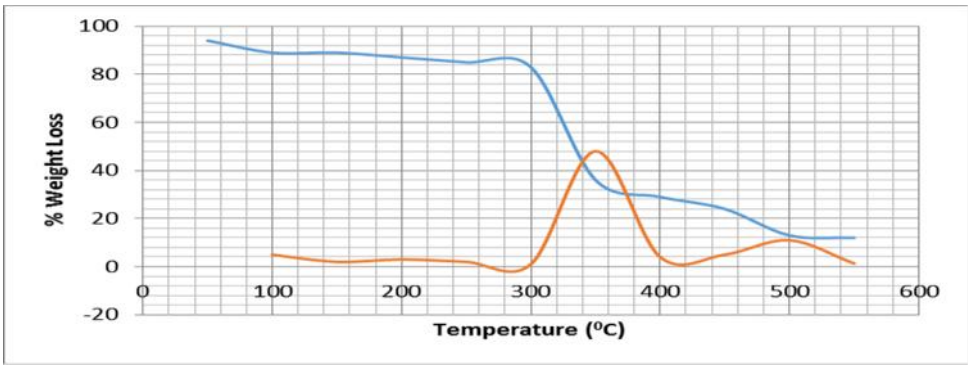

Figure 1: Typical thermograph and derivative thermograph of the extracted mucilage powder

Table 2: TGA Characterization of Some Plants Mucilage

Mucilage	Decomposition Step	Temperature range (^{0}C)	DTG peak (^{0}C)	Weight Loss (%)
CMP	1	50 – 100	200	10
	2	260 – 400	350	50
WMP	1	50 – 150	250	8
	2	260 – 380	350	56
APP	1	50 – 150	100	8
	2	250 – 400	350	55

Table 3: Thermal Stability Characterization of Some Plants' Mucilage

Mucilage	IDT (^{0}C)	Overall Loss (Mg)	Mucilage Residue
CMP	250	6.40	3.25
WMP	260	6.76	3.12
APP	260	6.61	3.27

Table 4: Degradation Kinetic Parameters of Mucilage Powder

Parameters	CMP	WMP	APP
Activation Energy (KJ/mol)	29.12	8.864	27.70
Frequency Factor (S^{-1})	0.032	0.082	1.492

Table 5: Elemental Characterization of Mucilage Powders

Mucilage	C (%)	H (%)	N (%)	S (mg/kg)	WC/N	Fe	Pb	Cr
CMP	70.04	24.70	2.59	757.8	26.02	3.08±0.08	0.04±0.01	0.05±0.01
WMP	73.65	23.86	4.40	930.8	16.72	16.06±0.34	0.13±0.03	0.13±0.03
APP	75.34	19.78	3.10	914.6	24.30	13.62±0.18	0.12±0.02	0.15±0.01

3.2 Discussions

The structure and functional properties of polysaccharide mucilage and gums influence their thermal stability and transition temperature generally, dehydration, deploymerization and pyrolitic processes were identified during heating and the TGA and DTG curves obtained are unique for a given polysaccharide. This implies that the curve can be used for identification of a particular polysaccharide mucilage powder

(Yiling, 2012). However the purity of such powder needs to be established for the thermrigravimetric method of identification to be reliable.

The TGA and DTG curves revealed the degradation of the polysaccharide at high temperature between 260 – 400^{o}C. However, because of the differences in structures and functional groups in the polysaccharides¸ the degradation routes or the resulting fragments will be different. Most polysaccharides compose of carboxylate or carboxylic acid functional groups hence, thermal degradation of the carboxylate groups and evolution of CO_2 from the corresponding carboxylate backbone may be the likely mechanism for the thermal transitions. However, assigning accurate transitions is very difficult (Brown, 2001).

The details of the thermal behaviour and thermal stability data according to the primary thermographs and derivatographs are shown in tables 2, 3 and 4. The initial weight loss in mucilage samples was attributed to the loss of adsorbed and structural water in the polysaccharide molecules due to desorption of moisture as hydrogen bound water molecule to the polysaccharide structure. The second weight loss may be linked to the polymer degradation (Sunil *et al*, 2012). The onset of weight lost represent onset of oxidation or decomposition and this indicates that the polysaccharide samples examined have good thermal stability (Yiling, 2012).

The elemental analysis indicates that the polysaccharide sample consist of certain percentage of carbon nitrogen, sulphur and hydrogen in CMP, WMM and APP which possibly suggest the presence of sulphur containing protein (Sunil *et al*. 2012).

FT-IR spectrometry has been widely used to characterize the polymer's molecular and material structure. FT-IR characterization often results in the identification of functional groups and their mode of attachment to polymer backbone (Sudarshan and Sunil, 2012). The FT-IR spectra exhibit the typical bands and peaks characteristic of polysaccharides. The peaks obtained at 3722 – 3286 cm^{-1} results from the presence of hydroxyl (-OH) group. Absorption bands around 1618 – 1430 cm^{-1} are typical of carboxylate groups of galacturonic acid residues. The region between 1500 – 1800 cm^{-1} are typical of carboxylic acid groups while absorption peaks at 1740 cm^{-1} and 1258 cm^{-1} are typical of acetyl group (Bharath *et al*, 2011). However, the absorption between 800 and 1200 cm^{-1} represents the fingerprint region for carbohydrates.

4.0 Conclusions

FTIR analysis has been used for the identification of mucilage powders especially the presence of different functional groups present. The TGA analysis as established that the powders are stable at high temperatures and this indicate that the powders can withstand high temperature operations involved in pharmaceutical industry such as tableting. Mucilaginous powders of plants' origin can be used in different industrial applications where high temperature operations are required. The abundance of these sustainable natural products can be explored in industrial applications because of their inherent advantages.

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