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Synthesis and characterization of carboxy methyle cellulose CMC from *Moriga Olievera* pods fruit

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Abstract
Cellulose pulp extracted from *Moringa oliefera* pods was characterized and used as raw material for preparation of carboxy methyl cellulose CMC. The physicochemical properties of CMC studied using FT-IR spectroscopy it was presence of a new and strong absorption band at 1606.59 cm^{-1} confirms the stretching vibration of carboxyl group (coo-), X-ray diffractometry XRD shows additional bands at 34.6Ɵ and 44.6Ɵ due to change in crystallinity by broadening or cleavage of hydrogen bonds by carbon methyl cellulose substitution at the hydroxyl groups of cellulose (Mondal, *et al* 2015),Thermal analysis TGA curve of CMC shows mass loss in between 250c and 300c due to loss of CO_2 from the polysacharideas a result of decarboxylation of COO- group, DTG maximum weight loss of dehydration of CMC is 43.11c while the weight loss of degrradtion is 102.33c less than the cellulose, DTA shows endothermic peaks at 81.72c due to decomposition of the main chain and depolymerisation (Biswal and Singh 2004).

Keywords: cellulose, carboxy methyl cellulose, *Moringa Oliefera* pods, carboxymethylation

Introduction
Many unwanted agricultural waste products or by-products from agricultural activities and agro-based processing litter the environment and constitute pollution. Most of the these agricultural wastes are composed of cellulose from plant cell walls. Cellulose is a linear and high molecular weight polymer which does not dissolve readily in water and organic solvents, making it unsuitable for most industrial uses. Cellulose is converted to useful chemical feedstock. carboxy methyl cellulose CMC is an important polymer derived from cellulose. CMC is a copolymer of two unites: β-D-glucose and β-D-glucopyranose 2-O-(carboxy methyl)-mono sodium salt, not randomly distributed along the marcromoleaule, which are linked via β-1,4 glycosidic bonds. The substitution of hydroxyl groups by the carboxy methyl is slightly preponderant at C-2 of the glucose (charpentier *et. al*., 1997). CMC is widely applied in a lot of industrial sectors including food, paper making, paints, pharmacetytics, cosmetics and mineral procrssing (Barbucci, magnani and consume, 2000; *et al*., 2009) due to it is simply and low cost to process. In addition it is used in other industries such as adhesives (Gayrish *et al*., 1989), Lubricants (soper, 1991), pesticides (Lee and FarreTorras, 1993) [3], Textiles (kniewske *et al*., 1999), Cements (Ernandes de Brito, 2000), paper (seiichi and shosuke, 2000; Tiitu *et al*., 2006), mango coating (Rachtanapun *et al*., 2008). CMC is obtained by activaition of the cellulose with aqueous NaOH is slurry of an aqueous organic solvent following reacting the cellulose with mono chloro acetic acid. The first step in carboxymethylation is an alkalization where first in carboxy groups of cellulose molecules are activated and changed into the more reactive alkaline form from CLL-O-)

$$\text{CLL-OH+NaOH} \longrightarrow \text{CELL-ONa+H2O}$$

This is followed by etherification in the sconed step (2)

$$\text{CLL-ONa+CL-CH2-CONa} \longrightarrow \text{CLL-O-CH2-COONa+NaCL}$$

Experimental
1. Materials
Moriga Oleifera pods were collected from a farm in north khartoum, washed, crashed grinded and mill sieved to suitable size and again washed and air deried. Were sodium hydroxide, mono chloro acetic acid, hydrochloric acid, ethanol, methanol, glacial acetic acid, iso propanol, sodium chlorite.All other chemicals are of analytical grade and used with out further purification. All aqueos solutions were prepared using deionized water.

2. Preparation of sample
2.1. Isolation of cellulose from *Moringa olifera* pods
In 1M 4% NaOH at 80c for 4 h. Then it was washed with distilled water to remove NaOH. The sconed sample was bleached with white clorox (containing 14% NaOCL). The bleaching steps were repeated four times and the products was washed several times with distilled water until the ordor of hypochlorite disppeared then dried at 100c.

3. Preparation of carboxy methyl cellulose
Carboxymethylation was carried out by suspending 5g of cellulose in 150 ml of iso propanol. 15 ml of 20, 25, 30, 35, 40, 45% NaOH with continous mechanical stirring over period of 30 minutes at room chloro acetic acid was added over aperiod of 30 min. the mixture was left with continous stirring for 3.5 hours at 55C After the end of the reaction the product precipitated by adding methanol followed by neutralization with 10% acetic acid filtered and washed with 70% ethanol the precipitate were washed with absolute methanol and deried at 60C.

Rusults and Discussion
1. FTIR analysis
The FTIR spectra of prepared CMC shows that the broad absorption band at 3417.63 cm^{-1} is due to the stretching frequency of – OH group. A band at 2937.38 cm is attributable to C-H stretching vibration. the presence of

anew and strong absorption band at 1606.59 cm confirms the stretching vibration of carboxyl group (coo), 1427.23cm is assigned to carboxyl groups as the sample saltes. The bonds 1290.29 cm and 1137.92 cm are assigned to – oh bending vibration and – C – O – C stretching, respectively. wavelength 850.55 cm is detected for 1,4 – β glycoside of cellulose.

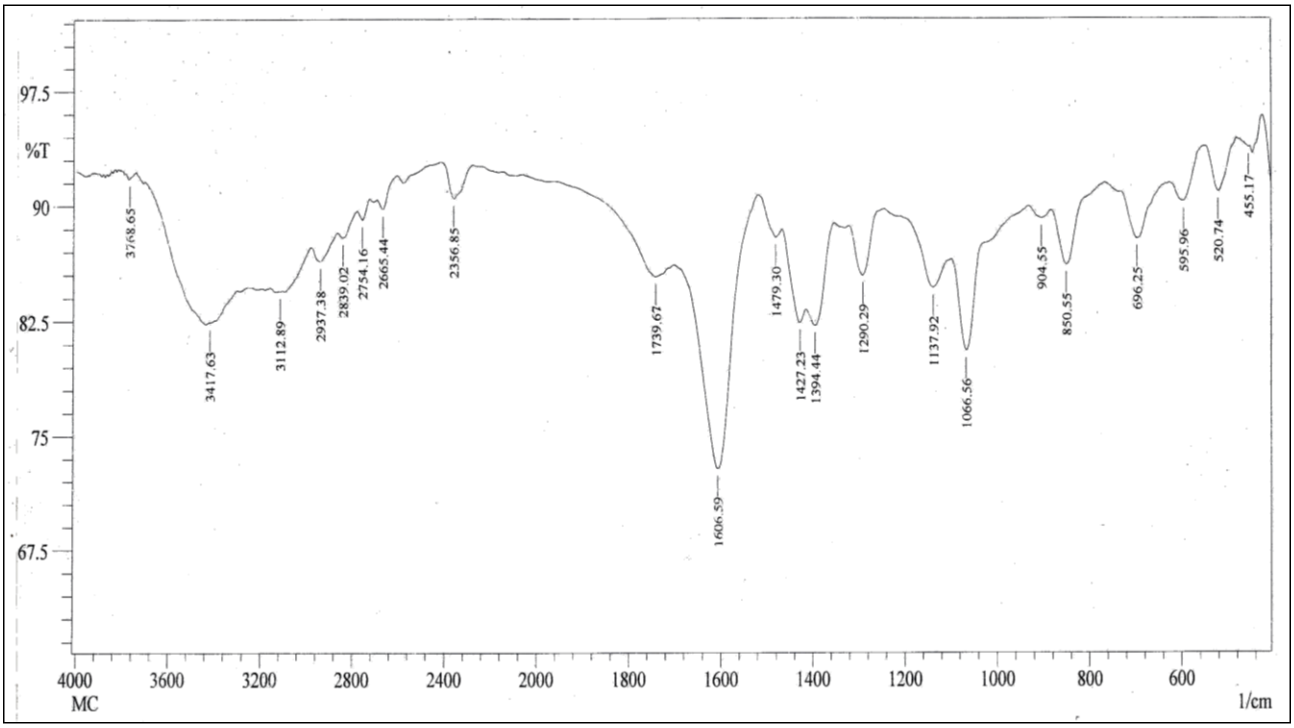

Fig 1: FTIR of CMC

2. XRD analysis

X-ray diffraction (XRD) pattern of CMC shows tow relatively in tense reflections at 8.3 θ and 20.1θ and additional bands at 34.6θ and 44.6θ. This phenomenon may be due to change in crystallinity by broadeing or cleavage of hydrogen bonds by carbon methyle cellulose substitution at the hydroxyl groups of cellulose (Mondal, *et al*., 2015) (Kumar *et al*., 2012) postulated that the 2θ=8 peak represents the degree of cellulose modification.

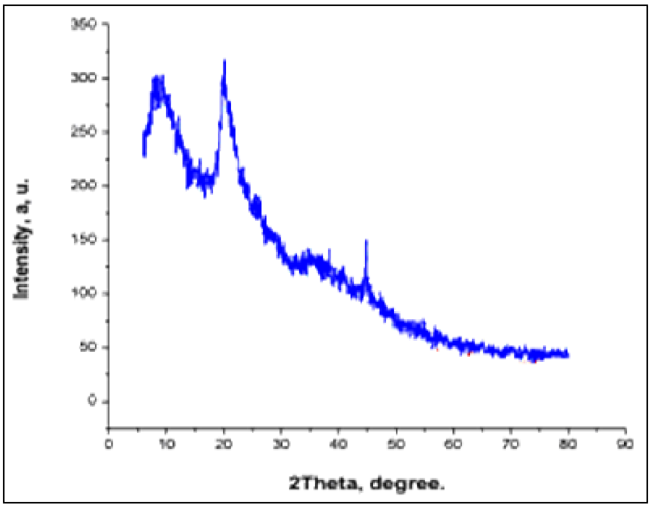

Fig 2: XRD spectrum patterns of CMC

3. Thermal analysis

Figure 3-A: shows that TGA curve of CMC. cellulose and cellulosic derivatives showed tow mass steps. the first at 47-100 C is associated with the loss of adsorbed water, while the second for cellulose is between 220C and 400C attributed to the actual pyrolysis and deccomposition for carboxy methyl cellulose (CMC), mass loss in between 250C and 300C due to the loss of CO_2 from the polysacharide as a result of de carboxylation of COO- group in this temperature range. The rate of weight loss is increased with increase in temperature (Biswal and Singh 2004).

Figure 3-B: shows DTG curves of carboxy methyl cellulose. The maximum weight loss of dehydration and degradtion states of cellulose happen at 67.48C and 30.45C respectively. Maximum weight loss of dehydration of CMC is 43.11C, while the weight loss of degrradations is 102.33C, less than that of cellulose (Biswal and Singh 2004).

Figure 3-C: shows the DTA thermal analysis of carboxy methyl cellulose. cellulose degrades immediately with one endothermic between 333.09 C to 400C 9 (Poletto *et al.* 2012). CMC showed endothermic peaks at 81.72C, due to decmposition of the main chain and depolymerisation, which proceeds due to the cleavage of glycosidic linkages. And exothermic peak at 312.57C due to the combustion of the degraded products (Biswal and Singh 2004).

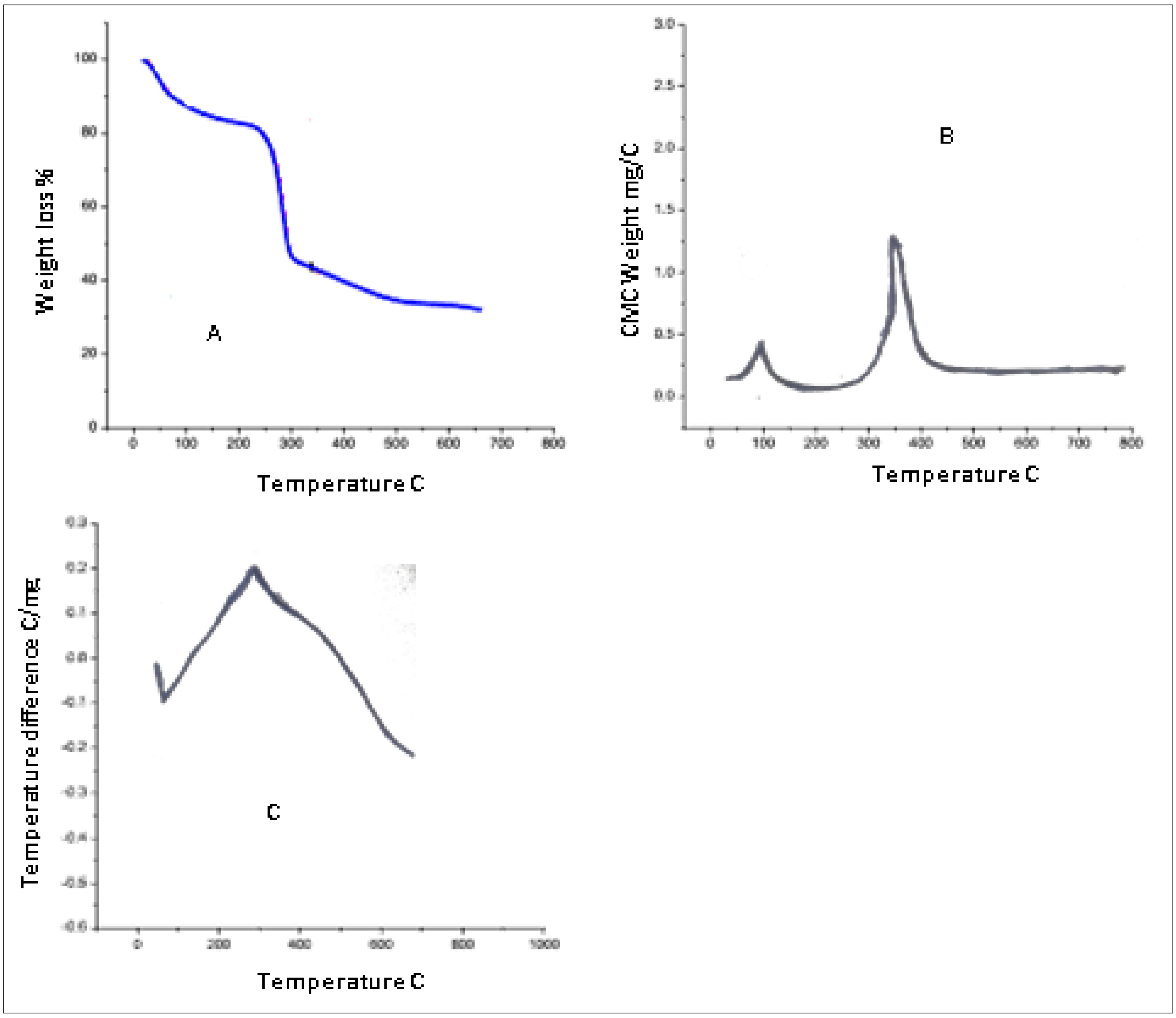

Fig 3: Thermal analysis curves (A) TGA, (B) DTG, (C) DTA of CMC

Conclution

α-cellulose was extracted from *moringa olievera* pods and etherification reaction was carried out by the infusion of cellulose with NaOH and mono chloro acetic acid. High purity food–grade CMC was successfully produced as an additive for pharmaceutial and food industries.

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