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Synthesis and characterization of methyl cellulose from moringa oleifera pods fruit

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Abstract
The synthesis of methyl cellulose (MS) from chemically purified cellulose extract from *Moringa Oleifera Pods* was carried out by base catalysis using 50% NaOH solution for 1 hour at room temperature. Excess NaOH was removed by filtration and Aceton was added. Di methyl sulfate (DMS) (50%,100%,150%,200%,250% and 300%) (v/m) based on cellulose dry weight was added drop-wise at 50C with constant stirring. After 1 hour of reaction the system was filtered and fresh reagents were added (acetone and DMS). The synthesized products were characterized by FTIR,H^1.NMR,C^{13}.NMR, XRD. synthezied methyl cellulose enhances the value to this abundant agro-industrial residue and extends its range of bio medical applications.

Keywords: cellulose, moringa oleifera fruit, methyl cellulose, base catalysis

Introduction
Moringa oleifera is a fast-growing, drought-resistant tree of the family *moringaceae*, it is widely cultivated for its seeds pods and leaves, used vegetables and traditional herbal medicine, it also used for water purification. Methyl cellulose (MC) a bio degradable polymer, is well known for its use as Inviromental friendly products, especially as coating or mulching film. methyl cellulose is one of the most important routes of cellulose derivatization and its one of the important cellulose ethers. Methyle cellulose presents an increased thermal stability, solibility in water associated in with an increased of degree of substitution (DS) which improves the commercial application of the polymer (Fillo *et al*. 2007). Several products of considerable commercial importance can be used as thickener in the Food industry (Reiber and conlin,1999) [4], as matricxfor controlled release of druges in pharmaceutical industry (mitchl *et al*., 1993), as admixture for concrete in civil construction activities depends on its solubility in water, and consequently, on the degree of substitution (DS) of the polymer. When this polymer is prepared with a DS between 1.4 and 2.0,cold or hot water may be used to produsesolutions or dispersions in low concertration (zohuriaan and shokrolahi, 2004). For DS higher than 2.0 the polymer shows solibility in organic solvents (Fillo *et al*., 2007). its synthesized methyl cellulose from *Moringa oleifera* pods cellulose with di methyl sulfate (DMS) in the presence of sodium hydroxide and acetone as solvent in heterogeneous conditions. Information structure and morphology was determind using Fourier transform infra-red spectroscopy (FTIR), X-ray diffractometry (XRD), proton nuclear magnetic resonace (H^1-NMR) and carbon -13 nuclear magnatic resonance (C^{13}- NMR).

Materials and Method
1. Materials
Moriga oleifera pods were collected from a farm in north Khartoum, washed, crashed, grinded and sieved and again washed and air deried. NaOH, aceone, Di methyl sulfate (DMS) acetic acid were of analytical grade. Distilled water was used through out the experiment.

2. Method
2.1. Isolation of chemically purified cellulose
Cellulose was isolated using 4% NaOH at 80C for 4h. It was then washed with distlled water to remove excess NaOH. 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 order of hypochlorite disappeared then dried at 60C.

3. Perpration of methyl cellulose
MC was prepared by base catalysis of (3.0 g) purified cellulose of *moringa oleifera* pods using 60 ml of 50% NaOH solution for 1 hour at room temperature. excess NaOH was removed by filtration. And aceton (27.0 ml) was added Dimethyl sulfate (DMS) (50%, 100%, 150%, 200%, 250%,300% v/w) based on weight of oven dried cellulose was added drop-wise at 50C with constant stirring. After 1 hour of reaction the system was filterd and fresh reagents were added (acetone, DMS). The same procedure was repeated for 2,3,4,5,and 6 hours reaction time. Methyl cellulose obtained was neutralized using acetic acid (10% v/v), filtered and washed with acetone (three times) and dried at 50C (Kumar and Walia 2014; oliveira *et al*. 2015) [3]. The degree of substitution (DS) of methyl cellulose was calculated (Bhatt *et al*. 2011; Kumar *et al*. 2012) [3].

Results and discussion
1. FTIR of methyle cellulose (MC)
FTIR of methyl cellulose was conducted using (Mattson 5000 FTIR spectrometer) in the rang of 4000-400cm (kumar *et al* 2012) [3].
Fig-1 depicts the spectrum of pure MC.
As can be seen pure MC had O-H starching vibrations at 3444.63 cm^{-1}. C-H stretching at 2923.88 and 2495.72 cm^{-1} C-O carbonyl stretching from glucose in the cellulose at 1764.75 cm^{-1} oferlaps adsorbed water. C-O stretching from asymmetric oxygen bridge at 1118.64 cm^{-1} and ring stretching at 877.55cm^{-1} are in the (fingerprint region). moreover, the spectrum also showed characteristic cellulose peaks in the rang 1000-1200 cm^{-1}. The 1118.64 cm^{-1} peak was ascribed to the asymmetric C-O-C stretch. The 1434.94 cm^{-1} peak corresponds to MC C-H bending.

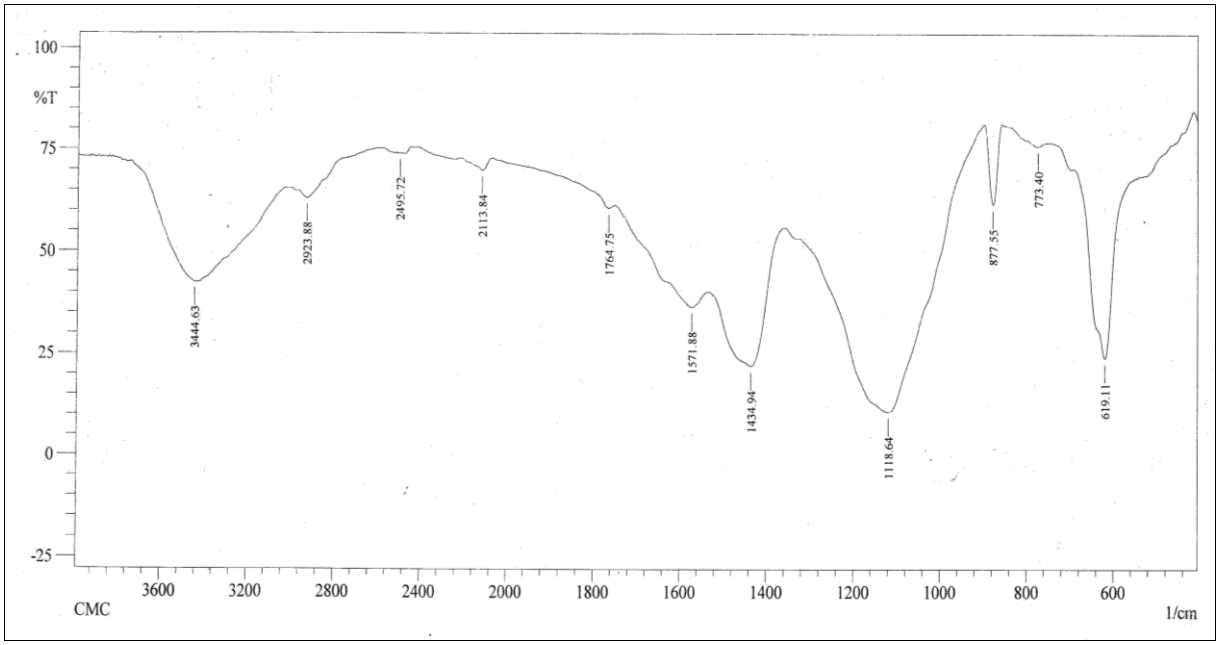

Fig 1: FTIR spectra of MC

2. Proton nuclear magnetic resonace (H^1 NMR)

H^1 NMR of methyl cellulose was conducted using Burker 400 NMR spectrometer equipped with dual probe(C13 and H1) in DMSO solvent (olivira *et al*.2015). H^1 NMR a powerful tool for evaluating the chemical structure of the compound in supporting the data of FTIR values of functional group. The signal number indicates the number of the proton position on the anhydroglucose unit. The proton signals of methyl groups at 2,3 and 6 position are labeled as 2,3 and 6Me. In H^1 NMR spectra of methyl cellulose in DMSO solvent, the proton signals overlap with each other because of the conformation changes of structre. The signals due to 2-Me and 3-Me at 3.58 ppm were not well distinguished while H-1 and 6-OH became single signal because of deuteration of three unsubstituated hydroxyle (-OH) groups (2-,3-, and 6-OH) of methyl cellulose into OD. The signals for H-4, H-6, H-1, H-3 and H-5 were appeared at 3.60 ppm, 3.72 ppm, and 3.88 ppm, 4.5 ppm, 3.4 -3.5 ppm, respectively. The signal of substituted methyl groups at the position in commercial methyl cellulose (randomly substituted methyl cellulose) are observed at 3.4 ppm (sekiguch *et al*., 2002) which supports our results.

C^{13} NMR of methyl cellulose shows four resonance lines divided into three different spectral ranges (85\75,60, and 40 ppm). carbons C2,C3,C4 and C5 signals appears in the double peak region at 85 and 75 ppm. However; if C6 is substituted these C6 resonance also fall into this spectral rang. Finally, the singlas of all unsubstiuted C6 carbons and of all methyl groups replaced in the methylation, compose the resonance line around 60 ppm.

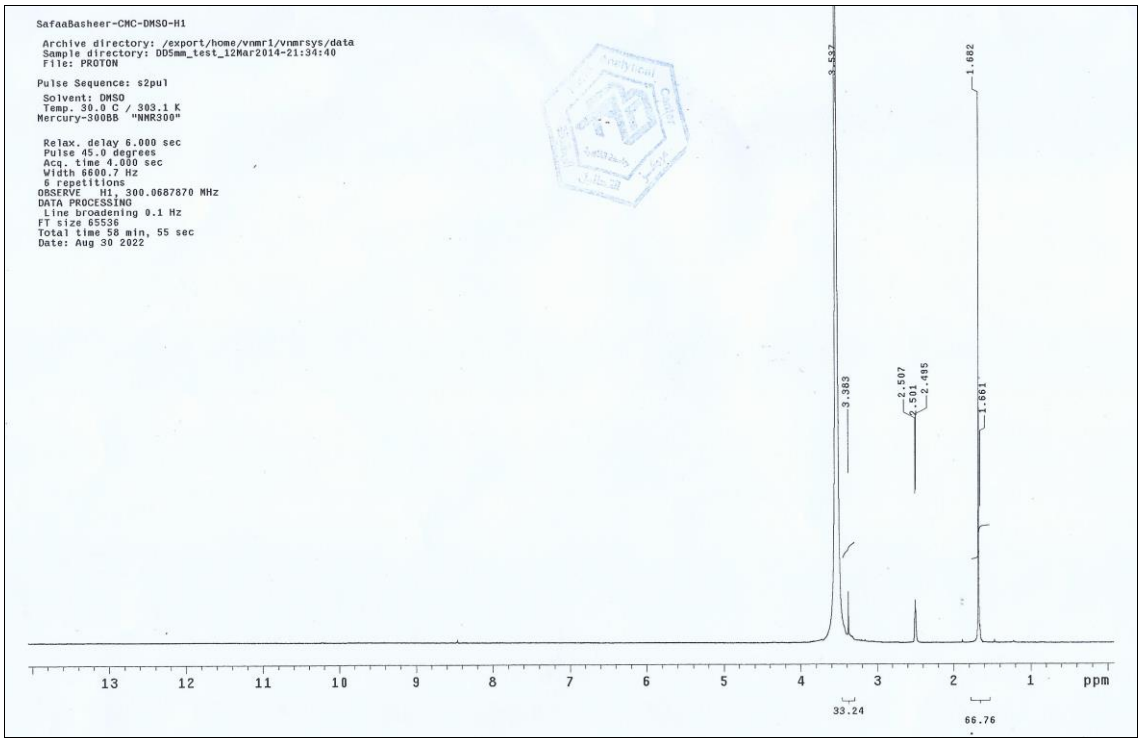

Fig 2: H^1NMR of MC

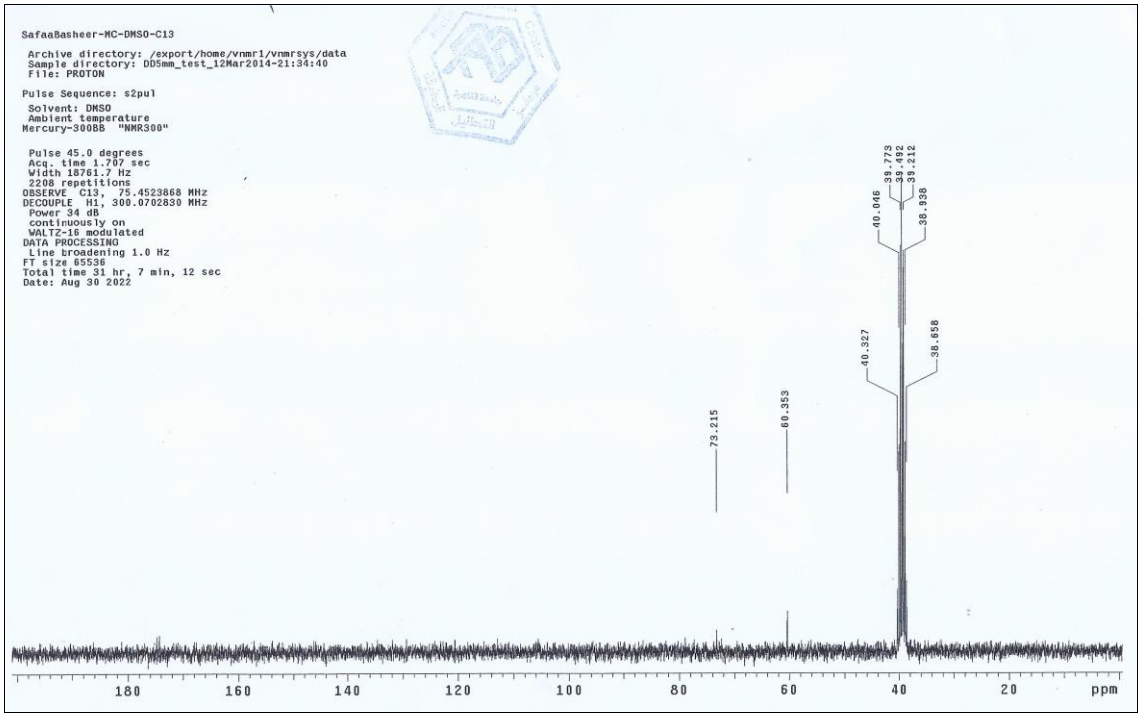

Fig 3: C^{13} NMR of MC

X-ray diffraction data of methyl cellulose

X-ray patterns were taken using Cuk radiation source by supplying 40 kv and 40 ma to X-ray generator. pure MC showed a semi crystalline structure. The patterns exhibited a sharp peak at 2θ=8.9 and abroad peak centered at 2θ=19.8 in a greement with pinotti *et al*. We ascribe the 8.9 peak to glucose-type crystalline order in the MC. Rimdusit *et al* concluded that peaks at 2θ=9.21 indicate the intermolecular structure of MC. Other postulated that the 20=8 peak represent the degree of cellulose modification.

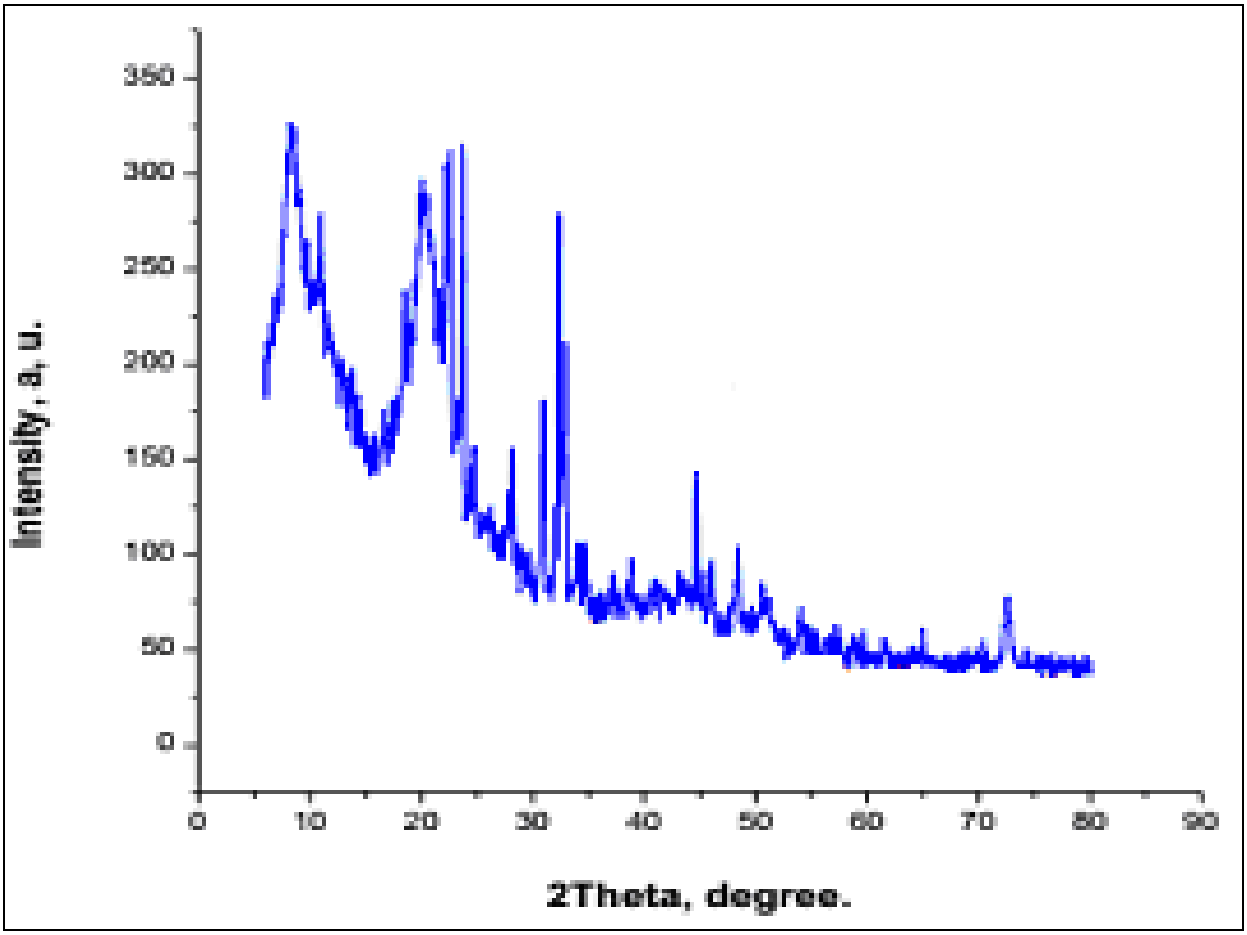

Fig 4: XRD` spectrum patterns of MC

Conclutions

Chemically purified cellulose was isolated from *moringa oleifera* pods and water soluble methyl cellulose was prepared by methylation of cellulose using di methyl sulfate (DMS) into acetone as solvent. FTIR data show the changes in structure due to the chemical treatments during synthesis and prove the modification of cellulose to methyl cellulose. H^1 NMR, C^{13} NMR confirmed the structure of the synthesized methyl cellulose. X-ray diffraction (XRD) studies show structural support for the structure of methyl cellulose with FTIR and H^1 NMR, C^{13} NMR data and the changes in crystallinity during chemical treatments from raw *moringa oliefira* pods to chemically purified cellulose, and synthesized methyl cellulose.

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