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Room Temperature Synthesis and Characterizations of Cu-MOF using Natural Polysaccharide as Potential Organic Linker

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Abstract: ***In this study, copper-based metal-organic framework was successfully synthesized using natural polysaccharide, gum Arabic (gA, Acacia Senegal) as potential organic linker by adopting the synthesis method from Misran et al. with some modifications. The copper-based metal-organic framework (Cu-MOF) was obtained from the combination of copper salt, natural polysaccharide, and terephthalic acid (H_2BDC) at various ratio. The synthesis process was done with the addition of triethylamine (TEA) as catalyst. X-ray Diffraction (XRD) pattern exhibited the presence of a prominent peak assigned to (200), (400), (511), (731), (773), and (882) crystal plane almost similar to the octahedral copper-based MOF-199. Field Emission Scanning Electron Microscopy (FESEM) shown an agglomeration and flaky particle. Energy Dispersive X-Ray Spectrometry (EDX) shown three main elements exist inside the Cu-MOF which is carbon (C), copper (Cu), and oxygen (O). This study showed the possibility of natural polysaccharide as new, non-toxic, water soluble linker. Thermogravimetric Analysis (TGA) analyses of as-synthesized Cu-MOF exhibited three different weight losses when heated to ca. 600^{o}C. The first mass decrease was due to loss of water content followed by the loss of organic moieties inside the Cu-MOF framework and the last was due to the collapse of MOF's structure leaving metal oxide as the final residue. The Fourier-Transform Infrared Spectroscopy (FTIR) spectra suggested strong absorption band at ca. 900 – 1200 cm^{-1} attributable to C-O bonding, at ca. 1045 cm^{-1} attributable to C-H bonding originating from gum Arabic, at ca. 1500 – 1600 cm^{-1} from the organic linker benzene ring, and at ca. 3200 – 3500 cm^{-1} from hydrogen bonding. These results suggested that natural polysaccharide of gum Arabic exhibited the possibility to become a new, non-toxic, renewable linker for the MOF materials and played a significant role in metal-organic framework formation.***

Keywords: ***Energy Dispersive X-Ray Spectrometry, Metal Organic Framework, Organic linker***

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I. INTRODUCTION

The greenhouse effects and global warming were caused by the huge amount of carbon dioxide (CO_2) and flue gases released by transportation, manufacturing factories and power plants. Carbon capture and storage (CCS) technology is one of the solutions that have received considerable attention nowadays to slow down the global warming [1]. CCS technology was divided into three basic processes namely post combustion, pre-combustion and oxy-fuel [2]. The first is post-combustion which separated the carbon dioxide gas from the flue gases. Pre-combustion technology allowed easy separation due to the pressure of gas. Oxy-fuel technology used pure oxygen with high density of gas for carbon dioxide separation. Based on these technologies, post-combustion was easier and cleaner method to be applied compared to others [3].

Besides, the growth of industrial activities caused the water sources to be polluted. The pollution came from the industries product waste such as paint, batteries, chemical, and fertilizer. Heavy metal produced from the waste cause physiological disorder to human and the aquatic life engendered [4]. In order to eliminate the contaminants from water source requires longer time and to completely eradicate them was a challenged. Therefore, metal-organic framework materials (MOFs) were one of the promising candidates to solve environmental issues [5].

Metal-organic framework (MOFs) are porous crystalline structure with large internal surface area discovered by Yaghi *et al*. and has received huge attention in the eye of researchers ever since. The pore size of MOFs can be controlled by changing the precursors and synthesis procedure [6]. Numerous possible combinations of the metal-organic framework structure can be done with the variety of metal oxide precursors as well as organic linker. The application of MOFs includes carbon capture and storage, heavy metal removal from water, biomedical, sensing, separation, catalysis, and fuel production [7], [2], [4]. There are several types of MOF's which is MOF-74, MOF-5, MOF-199, UiO-66, ZIF-8, and MIL-53 [5]. The properties and ability of MOFs were unique and diverse as we change the oxides and linkers. In particular, MOF-199 exhibited open metal sites while MOF-74 exhibited one-dimensional pore [8].

Copper-based MOF was also known as MOF-199, HKUST-1 and Cu-BTC with commercial name of basolite was widely used in the applications of water treatment to remove heavy metals as well as other water contaminants [4]. The morphology of Cu-MOF displayed an octahedral structure. Cu-MOF demonstrated high potential toward gas storage and capture due to the porosity, high surface area and easy to synthesis [9]. There were several known procedures to synthesize Cu-MOF which were conventional hydrothermal, mechanochemical, sonochemical, and microwave-assisted method [10]. Conventional method was the standard method to synthesize MOF which the process for reaction time from several hours to days. Mechanochemical method allowed syntheses to occur with the absent of solvent. Sonochemical method used ultrasonic wave which allowed fast heating and cooling. Microwave-assisted method allowed researcher to manipulate power output for heating temperature at certain time. Thus, the resulting relative crystallinity and morphology produced were varied depending on the synthesis methods.

The general method to synthesize copper-based MOF involved copper salt was combined with 1, 3, 5-benzene tricarboxylic acid (BTC) or 1, 4-benzene dicarboxylic acid (H_2BDC) as the organic linker. These types of linker were known to be having carcinogenic properties that potentially harmful to human body. Thus, some researcher has used bio-based, renewable, non-toxic and sustainable materials for organic linker such as Cyclodextrin and peptide. Cyclodextrin knowns asα-CD,β-CD andγ-CD was synthesized by Zhang *et al*. using microwave technique. CD-MOF also can be syntheses using vapor diffusion method with small amount of reactant. CD-MOF has unique characteristic such as high surface area, porosity, and tunability. CD-MOF was used to increase the efficiency of biocompatible drug delivery due to non-toxic and edible properties [11]. Meanwhile, peptide is compound that link several amino acids in a chain. Peptide-based BioMOFs was commonly synthesize using dipeptides compared to polypeptides. The flexibility of peptide-based BioMOFs shown a huge potential toward molecular switches [12].

Natural polysaccharide, gum Arabic (*Acacia Senegal*) mainly obtained from Sudan was used as a thickening agent and an emulsifier in food. 1,3-linked β-D-galactopyranosyl were the backbone of gum Arabic [13]. The mineral content of gum Arabic are calcium (Ca), magnesium (mg) and potassium (P). The sugar content of gum Arabic consist of 38 % of galactose, 46 % of arabinose, 4 % of rhamnose and 6.5 % of glucuronic acid [14]. The ash, nitrogen and protein content were at ca. range of 3.6-4 %, 0.147-0.175 %, and 0.94-1.15 % respectively with a 13 cm/g of viscosity [13]. This organic material has the potential to synthesize with copper metal as the new linker.

Thus, in this study, the amount of 1, 4-benzene dicarboxylic acid (H_2BDC) used as the conventional organic linker was reduced with the addition of gum Arabic. The copper-based metal-organic framework was synthesized using natural polysaccharide, gum Arabic (gA, *Acacia Senegal*) as potential organic linker by adopting the synthesis method from Misran *et al*. with some modifications.

II. EXPERIMENTAL METHODS

Materials

The procedure to synthesize Cu-based MOF using natural polysaccharide as the new organic linker were done by adopting method from Misran *et al*. [15]. Copper nitrate trihydrate ($Cu(NO_3)_2.3H_2O$, HmbG Chemicals 99.5 %) from HmbG was used as the metal precursor. Terephthalic acid (1, 4-benzenedicarboxylic acid, H_2BDC, Merck, 98 %) from Merck and natural polysaccharide, consumer grade gum Arabic (gA, *Acacia Senegal*, $C_{24}H_{32}O_5$) were used as the linker, ethanol (synthesis grade, HmbG Chemical, C_2H_5OH), triethylamine (TEA, Merck, 99 %, $C_6H_{15}N$). All chemicals were used as purchased.

Synthesis Method

In this study, four factors namely TEA (catalyst) ratio, aging time, stirring time and gum Arabic content were chosen as the parameters to observe their effects on formation of Cu-MOF during the synthesis. Copper (II) nitrate trihydrate was dissolved in Millipore water to form the first solution (metal ions). Then, natural polysaccharide was also dissolved in Millipore water at room temperature to form the second solution (linker). Different amount of ethanol and terephthalic acid (H_2BDC) was added into the second solution until fully dissolved. Both the first and second solution was added together. The process proceeded with and without the addition of triethylamine (TEA) as catalyst. The mixture was continuously stirred using magnetic stirrer for certain hours and aged in the drying oven at ca. 313 K for 24 hours. The mixture was centrifuged and washed with Millipore water for several times. The light blue precipitate obtained were dried overnight at 313 K to yield dark blue powder as-synthesized Cu-MOF. This processing steps were repeated with different parameters, Cu-MOF 1 (No TEA, Aging, 24 hours), Cu-MOF 2 (TEA, No Aging, 24 hours), Cu-MOF 3 (TEA, Aging, 24 hours), Cu-MOF 4 (TEA, Aging, 12 hours), and Cu-MOF 5 (TEA, Aging, 12 hours, 23 % additional gum Arabic). Fig. 1 shows the flowchart of synthesized Cu-MOF using natural polysaccharide.

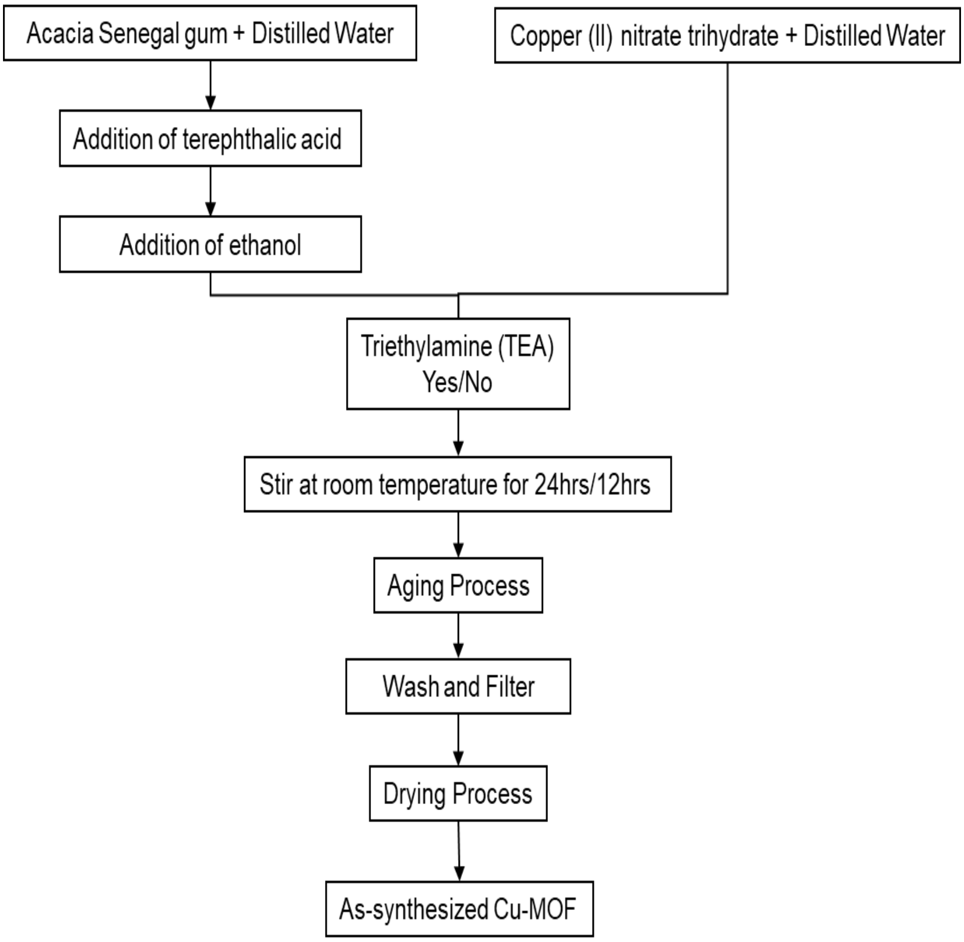


Fig. 1 Synthesis flowchart of MOF-199 using natural polysaccharide, gum Arabic (gA, Acacia Senegal) as an organic linker

Structural Characterizations

The as-synthesized blue powdery Cu-MOF were characterized by X-ray diffraction analyses (XRD), field emission scanning electron microscopy (FE-SEM), energy dispersive X-Ray spectrometry (EDX), thermogravimetric analysis (TGA), and Fourier-Transform infrared spectroscopy (FTIR). XRD patterns were obtained from Shimadzu XRD-6000 from 5°- 45° at 30 kV of voltage. The current, scan step and scan speed applied were 20 mA, 0.02° and 3°/ min respectively with CuK$_\alpha$ radiation of λ= 1.5406 Å [9]. The morphology of Cu-MOF with different parameters were observed from FESEM images. SU8030 Hitachi captured a sharp image using back-scattered electron (BSE) and secondary electron (SE) detector. These images were obtained when the accelerating voltage was at ca. 1 kV, 50k -100k magnification and depth of field at ca. 500 nm - 1000 nm [15]. The EDX analyses were done using the same instrument and parameter as the FESEM at two different locations using the images obtained from FESEM analyses. TGA were done using Netzsch TG 200 F3 Tarsus. The samples were heated at room temperature until it reaches 873 K with heating rate of 283 K per minutes and gas flow rate of nitrogen gas at 20 ml per minutes [16]. The Fourier-Transform Infrared Spectroscopy (FTIR) patterns were obtained from IRPrestige-21 Shimadzu model at 500 cm^{-1}-4000 cm^{-1} with resolutions number of two and 20 number of scans [17].

III. RESULTS AND DISCUSSION

The as-synthesized Cu-MOF were obtained as blue-powder were observed after drying. Colour concentration of Cu-MOF powder were observed when the synthesis parameters were changed. Red, blue and green (RGB) colour model were used to identified the colour concentration. Cu-MOF 1 produced a light mint green colour, Cu-MOF 2 produced light blue colour, Cu-MOF 3 produced a brown colour, Cu-MOF 4 produced a light blue colour, and Cu-MOF 5 produced a blue colour. The addition of TEA as the catalyst increased the colour concentration of the Cu-MOF from light mint to blue. The aging process with 24 hours of stirring time resulted in Cu-MOF with brown colour powder. There was no significant difference in Cu-MOF when the stirring time was reduced to 12 hours. However, higher amount of gum Arabic with less stirring time produced darker blue colour concentration. Thus, optical observation suggested the successful formation of Cu-MOF almost similar to MOF-199. Fig. 2 shows the X-ray diffraction (XRD) patterns obtained from the as-synthesized Cu-MOF exhibited six prominent reflection peaks compared to MOF-199 in previous studies which exhibited twelve prominent reflection peaks. The peaks were attributable to (200), (400), (511), (731), (773), and

(882) crystal planes assigned to octahedral structure [18]. Cu-MOF 1 without the addition of TEA exhibited highest peak at ca. $2\theta = 17.5°$, 25.9° and 28.4° compared to other samples of Cu-MOF 2, Cu-MOF 3, and Cu-MOF 5 suggesting highest relative crystallinity [19]. The peaks observed at ca. $2\theta = 17.5°$and 25.9° corresponding to (511) and (731) crystal planes. The presence of copper oxide (CuO) was observed at ca. $2\theta = 35.5°$and 39.6° corresponding to (773) and (882) crystal planes [18]. The major reflection peaks corresponding to (222) planes from the octahedral structure was not presence at ca. $2\theta = 11.7°$ for all samples prepared with and without TEA, the results suggested that the structure were not octahedral single crystal. On the other hand, other reflection peaks attributable to octahedral crystal structure of conventional MOF-199 were observed.

In addition, the impurities of isolated pure Cu_2O compound were not observed due to absence of reflection peak at ca. $2\theta = 36.43°$. These results suggested that the Cu^{2+} ions had successfully formed copper oxide clusters in the metal-organic framework [20]. Hence, the results suggested successful formation of Cu-MOF even with the addition of various amount of gum Arabic as new organic linker together with commercial linker.

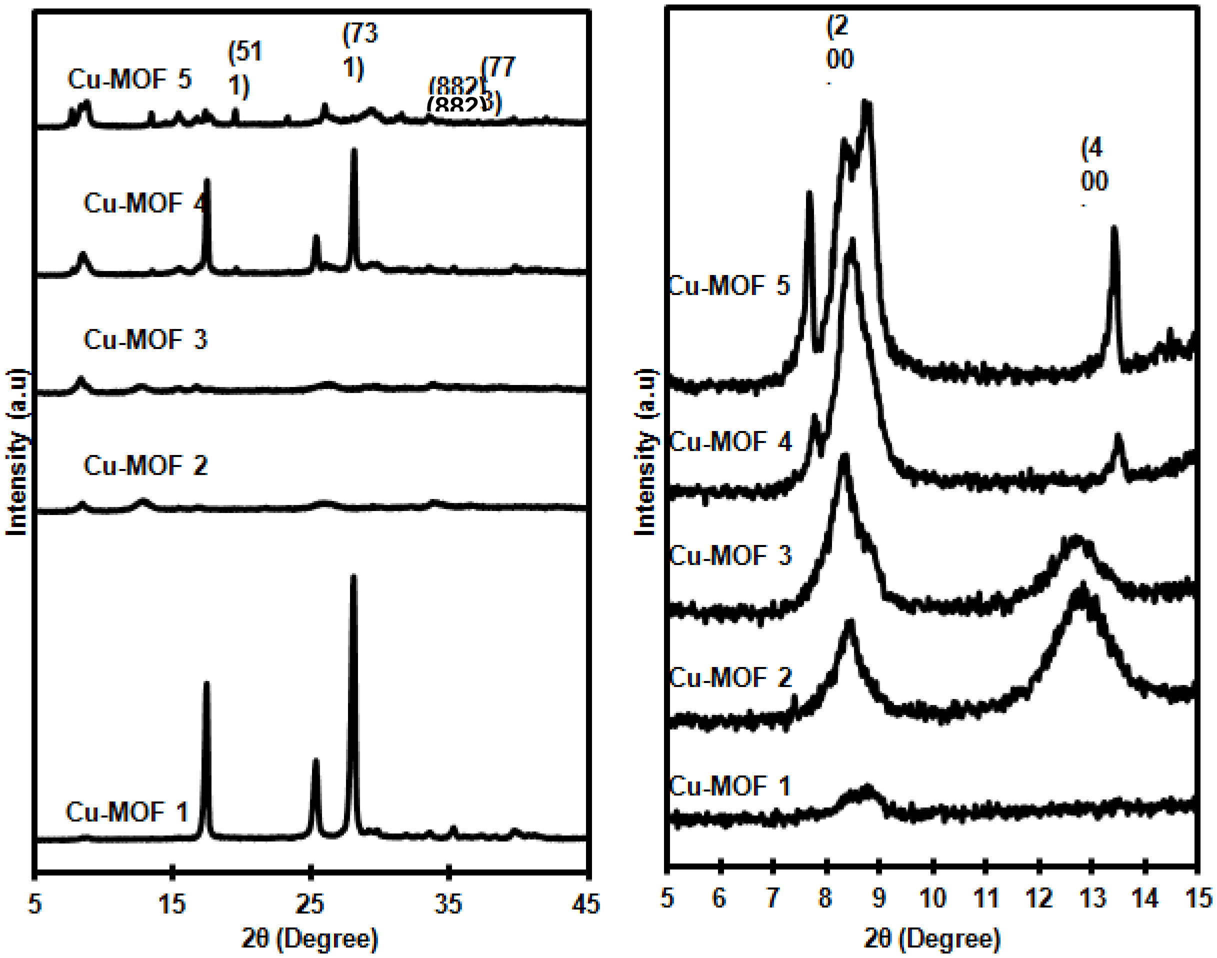


Fig. 2 X-ray diffraction (XRD) patterns of as-synthesized, dried Cu-MOF at $2\theta = 5°$- 45°and 5°-15°, relatively

Fig. 3 shows the morphology of Cu-MOF prepared with and without TEA as well as other parameters. Based on previous research done by Yaghi *et al.*, Chui *et al.*, and Misran *et al.* standard Cu-MOF (i.e. MOF-199) that were synthesized with 1, 3, 5-benzene tricarboxylic acid (BTC) or 1, 4-benzene dicarboxylic acid (H_2BDC) as the linker showed the presence of well-defined octahedral structure. In this study, the use of natural polysaccharide form gum Arabic, as new, non-toxic, water soluble organic linker in Cu-MOF formation exhibited different outcomes than the conventional structure. The morphology of all samples exhibited agglomerations and flaky particles. The octahedral structured was not obtained in this study suggested that the incorporation of gum Arabic into the framework of conventional organic linkers resulted in a flexible structure hence the flaky agglomerations observed [9].

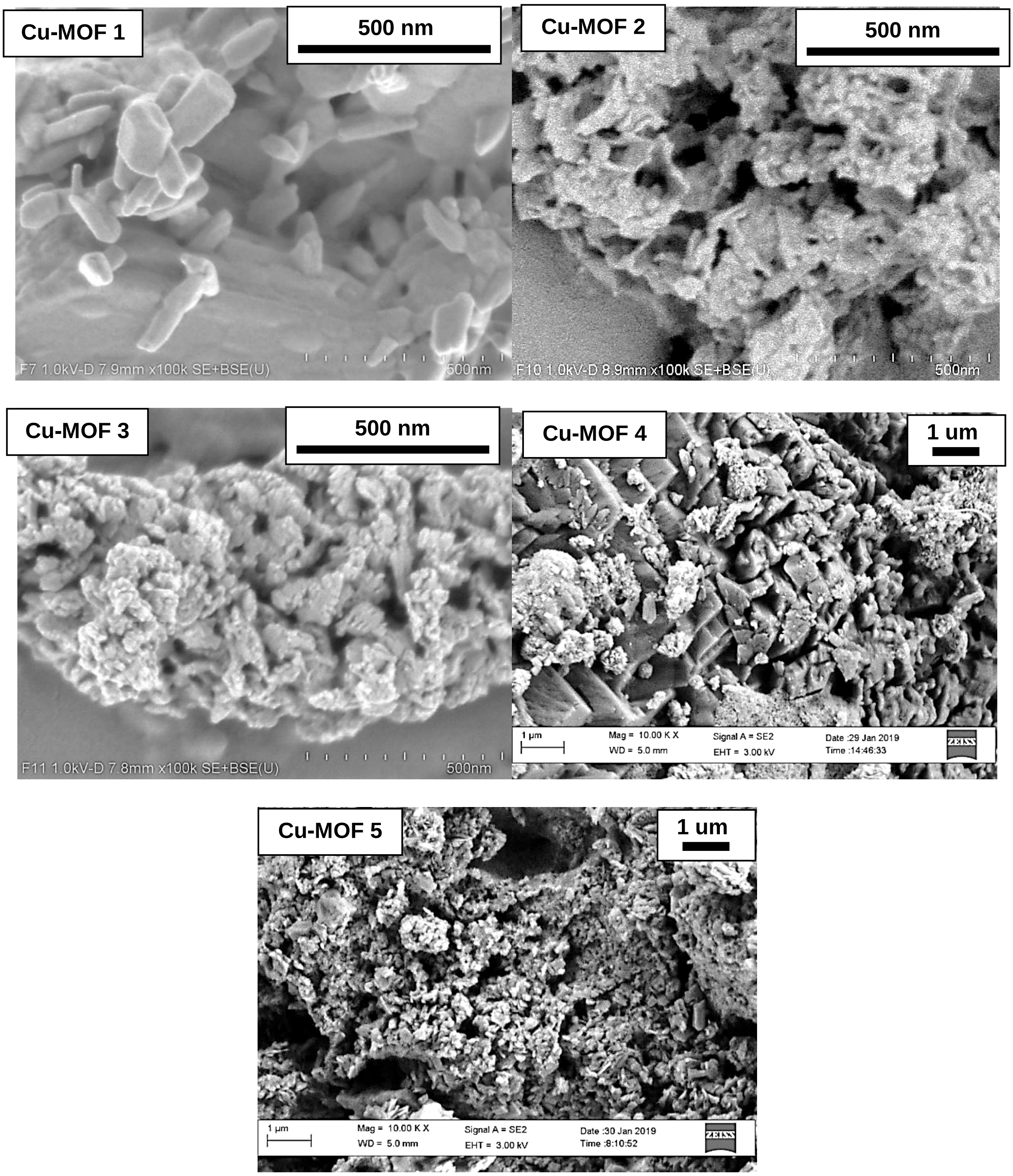
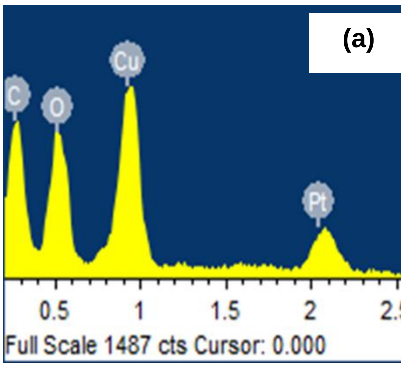
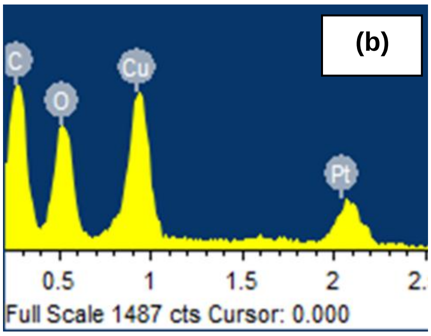


Fig. 3 FESEM images for as-synthesized, dried Cu-MOF produced using gum Arabic with and without TEA

Fig. 4 and Fig. 5 shows the EDX representative spectra for Cu-MOF 2 and Cu-MOF 4 at two different points. Elemental peaks attributable to copper (Cu), carbon (C), oxygen (O) elements were observed suggesting the successful formation of Cu-MOF with addition of gum Arabic as the organic linker. The weight percentage of carbon for Cu-MOF 2 was at ca. 20.3 % and 24.1 %, oxygen was at ca. 16.0% and 15.2 %, copper was at ca. 36.8 % and 31.9 % at first and second location, respectively. While, the weight percentage of carbon for Cu-MOF 4 was at ca. 38.0 % and 38.0 %, oxygen was at ca. 34.6 % and 36.3 %, copper was at ca. 27.4 % and 25.7 % at first and second location, respectively. The differences in weight percentage for both locations were suggested to be due to surface gradient. The Pt peak was originated from coating of non-conductive samples. Copper element exhibited in the EDX analyses suggested successful formation of metal cluster originated from copper (II) nitrate trihydrate. Carbon and oxygen were obtained from the organic linker which were gum Arabic as well as original linker.

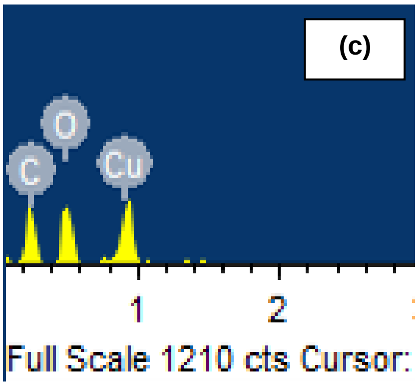


Sample	Cu-MOF 2	
Element	Weight %	Atomic %
C	20.29	49.58
O	16.02	29.38
Cu	36.77	16.99
Pt	26.77	4.05

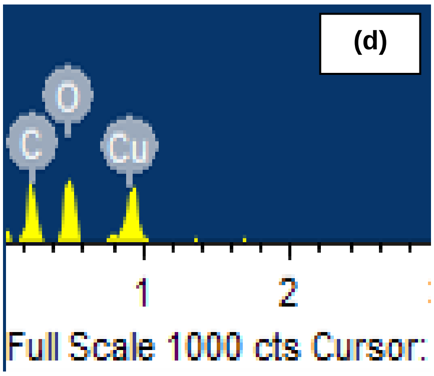


Sample	Cu-MOF 2	
Element	Weight %	Atomic %
C	24.07	55.61
O	15.19	26.35
Cu	31.89	13.93
Pt	28.85	4.10

Fig. 4 EDX representative for Cu-MOF 2 at two different areas



Sample	Cu-MOF 4	
Element	Weight %	Atomic %
C	38.03	54.98
O	34.59	37.54
Cu	27.37	7.48



Sample	Cu-MOF 4	
Element	Weight %	Atomic %
C	37.98	54.17
O	36.34	38.91
Cu	25.67	6.92

Fig. 5 EDX representative for Cu-MOF 4 at two different areas

The TGA curve of Cu-MOF 1 sample prepared without TEA in Fig. 6 shows a sudden drop in weight % at ca. 89.6 % when the temperature reached 240.1 °C. The sudden drop suggested structure collapse in copper and organic bonding as well as burning off organic linker of gum Arabic and BDC. After that, the weight loss of Cu-MOF 1 samples became plateau when the temperature reached ca. 589.9°C with a residual mass of ca. 8.40 %. Copper oxide (CuO) was the compound formed at the end of heating process that was stable making the weight loss curve constant until 600 °C. [18]. Cu-MOF 2 and Cu-MOF 3 exhibited three steps of weight losses. The first step occurred when the temperature for Cu-MOF 2 was at ca. 191.7 °C with ca. 26.53 % weight losses and temperature for Cu-MOF 3 reached ca. 191.1 °C with ca. 20.47 % weight losses. The first weight losses indicated the losses of physisorbed water contents, i.e. moisture in the samples [20].

The losses of organic moieties and the collapse of organic linker inside the Cu-MOF occurred in the last two steps [21]. Decomposition of Cu-MOF 2 and Cu-MOF 5 continue until the curve reached a plateau at ca. 598.9°C and at ca. 599.0°C with residual masses of ca. 40.58 % and ca. 42.12 %. The three-steps weight loss profile of TGA curves for samples prepared with the addition TEA during the synthesis were similar to those obtained for conventional MOF-199. These results suggested the successful formation of a new framework structure containing conventional commercial linker and gum Arabic.

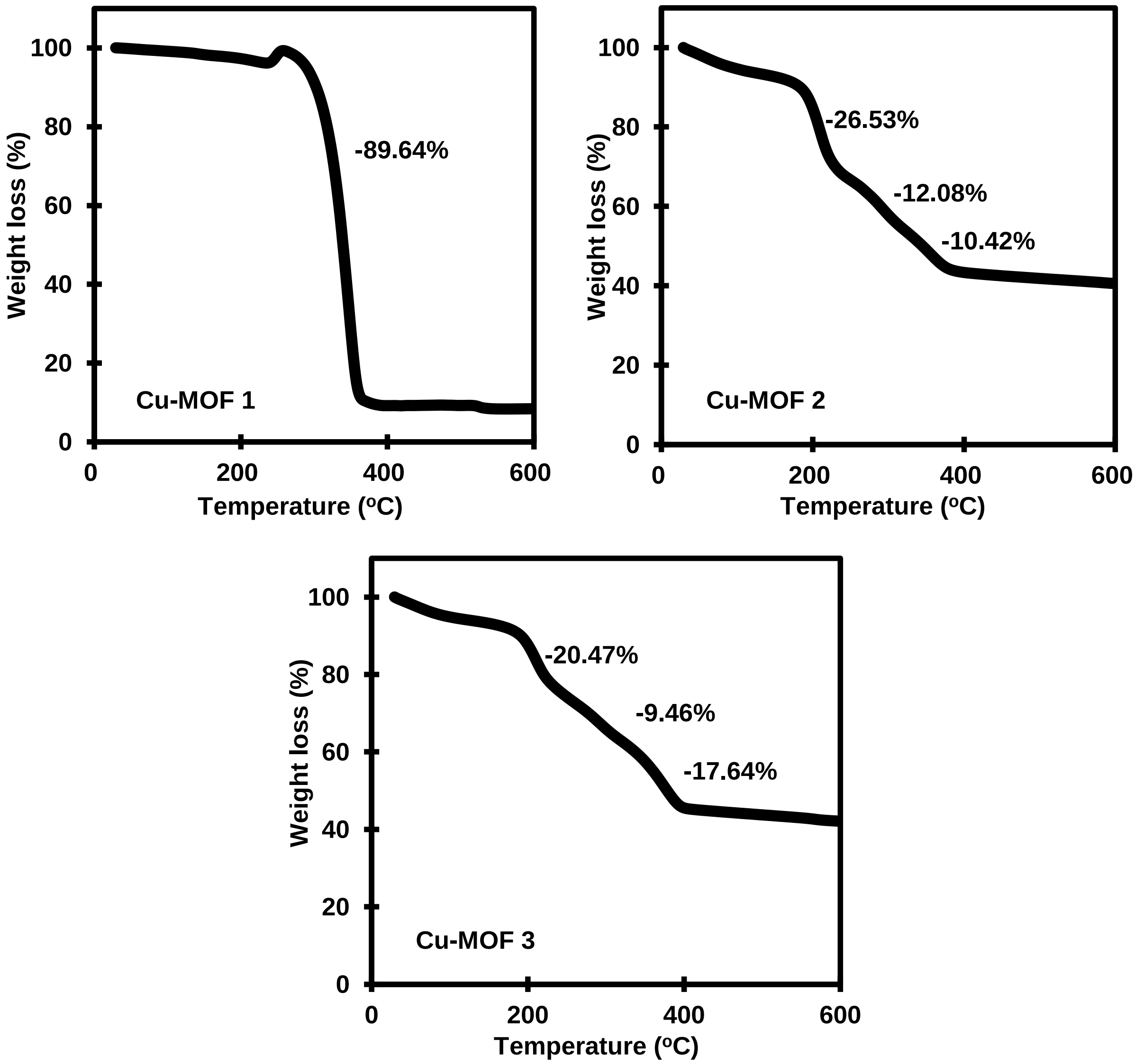


Fig. 6 Thermogravimetric analyses for as-synthesized, dried Cu-MOF prepared with and without TEA

The Fourier-Transform Infrared Spectroscopy (FTIR) spectra exhibited strong absorption band at wavelength of ca. 500 -1700 cm^{-1} and ca. 2700 - 3500 cm^{-1}. The broad absorption peaks cantered at ca. 600 cm^{-1} was assigned to metal-oxygen bonding vibration and at ca. 1500 – 1600 cm^{-1} from the benzene ring in the commercial organic linker [22]. At wavelength of ca. 1270 cm^{-1} and ca. 1375 cm^{-1}, absorption peaks from C-O bonding and C=O bonding, respectively were from the carboxylic acid presence in the gum Arabic inside the framework. The absorption band for C=C bonding was at ca. 1100 cm^{-1} [23], [24]. The natural polysaccharide shown their presence as C-H and N-H bonding vibration at ca. 1045 cm-1 and 1640 cm^{-1} respectively [25]. The bonding vibration occurred at high wavelength of ca. 3200 – 3500 cm^{-1} were from hydrogen bonding since the sample was dispersed in water as a medium during FTIR measurements [22], [26].

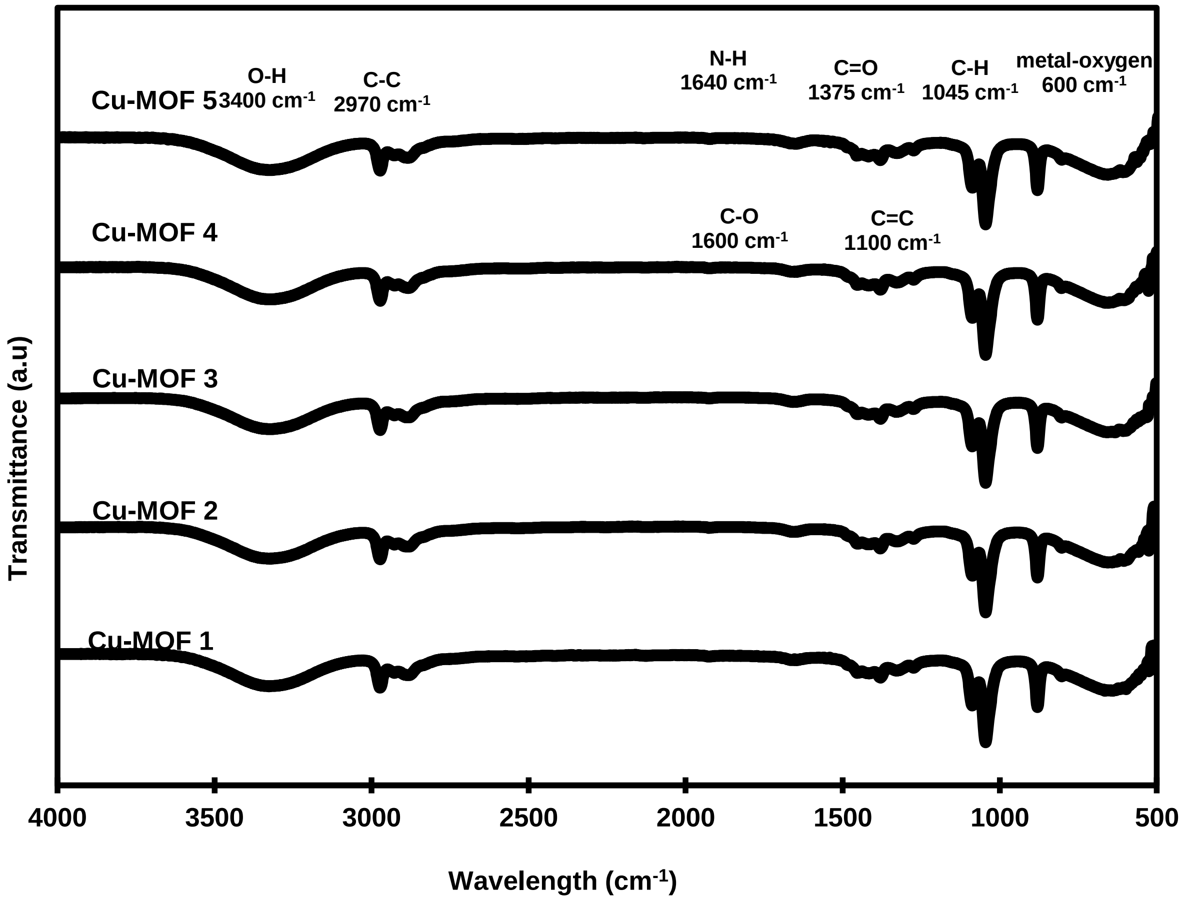


Fig. 7 FTIR spectra of as-synthesized, dried Cu-MOF prepared with gum Arabic and commercial organic linker with and without the addition of TEA as catalyst

IV. CONCLUSIONS

This study has shown the possibility of natural polysaccharide of gum Arabic as a new, sustainable, non-toxic and water-soluble organic linker. As-synthesized, dried Cu-MOF produced different colour concentration based on the RGB model depending on the new linker and catalyst concentration. Analyses results from structural characterizations suggested the presence of gum Arabic as the organic linker in the metal-organic framework. Cu-MOF were successfully formed, based on the losses of organic materials and the collapse of the organic linker in TGA results suggested similar weight loss curve similar to those obtained in copper-based MOF-199.The absorption peaks of natural polysaccharide was observed at 1600 cm^{-1} of C-H bonding vibration. In conclusion, the natural polysaccharide was successfully combined with copper (II) nitrate trihydrate to form water-soluble, less toxic Cu-MOF materials.

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