# Thermal and Morphological Properties of Polyvinyl Alcohol-based Hydrogel Containing Microcrystal Cellulose

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Abstract: In this work, the PVA-based hydrogels containing various loading of microcrystal cellulose (MCC: 0; 0.2; 0.4; 0.6 and 0.8) g were prepared in a bench-scale reflux-reactor using water as a solvent in an optimized condition. Other constituents added, i.e. acrylic acid (AA) and N'N-Methylene bisacrylamide (MBA) as comonomers as well as ammonium persulphate ((NH4)2S2O8) as initiator was keeping their composition ratio constant. Products of the interpenetrating-hydrogels were cast in plastic mold and cooled, and then characterized. First of all, their absorption properties and were measured using swelling test to know optimum conditions and the next will be characterized with FTIR, DSC, and SEM. Results showed that the optimum composition ratio of PVA/AA/MCC/MBA/APS = 0.4/4/0.6/0.06/0.2 enhanced the water absorption. FTIR analysis of the film specimen after exhaustive extraction in n-hexane still contained stable AA-carbonyl (C=O) absorption peak at 1713 cm<sup>-1</sup> of hydrogel 1 and 1707 cm<sup>-1</sup> of hydrogel 4. The thermal properties of the optimized composition of the hydrogel 1 showed its stable decomposition temperature (thermal stability of 482.28°C). Morphological properties of the interpenetrating-hydrogel micro composites also showed finely distributed of the micro filler, which is responsible for its improved mechanical and

# thermal properties.

# **1 INTRODUCTION**

Technological progress continues to increase. To improve research in the field of polymers, new technologies such as biopolymers are needed. Biopolymers are one of the materials produced by modifying a polymer. Modifications can be made by combining polymers that function to improve the properties of these polymers such as absorption, elasticity, and mechanical strength that are biodegradable, biocompatible, and non-toxic. This modification can also affect the solubility of polymers which can dissolve in water to be insoluble like hydrogels (Sinha, 2018)).

Hydrogels are hydrophilic polymers with threedimensional structures that have cross bonds (Ahmed, 2015). The three-dimensional structure of the hydrogel formed through crosslinking makes the hydrogel capable of absorbing and releasing water reversibly (Ambrosio, Demitri, & Sannino, 2011). The ability of a hydrogel to absorb water thousands of times from its dry weight is influenced by a group of hydrophilic functions found in three-dimensional structures. This hydrophilic functional group can hold large amounts of water (Ahmed, 2015, Maitra & Shukla, 2014). The hydrophilic functional groups contained in hydrogels such as carboxyl (-COOH), hydroxyl (-OH), and amide (-CONH<sub>2</sub>) (Ha et al., 2018). Hydrogels have several very important properties, which are able to expand well in water, insoluble in water (Burdick & Stevens, 2005), softness, elasticity, and flexibility. Because of these properties, hydrogels can be used in various applications for food, agriculture, industry, medical, medicine, and cosmetics (Kiatkamjornwong, 2007, Anamica & P. P. Pande, 2017).

At present, a lot of research has been done regarding the manufacture of hydrogels. Making hydrogels that use polysaccharides such as starch, chitosan, xanthan, and cellulose can be done through polymerization reactions. The use of chitosan polysaccharides with acrylic monomers is very

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efficient in making hydrogels (Mahdavinia, Zohuriaan-Mehr, & Pourjavadi, 2004). Initially, the hydrogel was made from cellulose and polyvinyl alcohol which have a hydrophilic group and have a high affinity for water. This type of hydrogel has several disadvantages including absorption capacity is relatively small, less stable to changes in pH, temperature and physical properties that are not good (Swantomo, Megasari, & Saptaaji, 2008).

The manufacture of polyvinyl alcohol (PVA) and polyacrylic acid (PAA) hydrogels with xanthan gum polysaccharides which can be applied as drug delivery control through cross-linked polymerization using N'N-methylene bisacrylamide (MBA) as a crosslinking agent and ammonium persulfate (APS) as the initiator. The best water absorption results are shown in hydrogels containing low MBA, namely 0.010 g (Bhattacharya et al., 2012).

Polyvinyl alcohol (PVA) is one of the most promising synthetic polymers for the development of biomaterials (Teodorescu, Bercea, & Morariu, 2018). PVA is a water-soluble polymer, easily degraded (Chiellini, Corti, D'Antone, & Solaro, 2003), has high tensile strength and flexibility, elastic, non-toxic, and biocompatible polymer. So it can be used in many applications make PVA a hydrogel which is widely used in the textile, adhesive, food, medicine, paper, packaging, and cosmetics industries (Peresin et al., 2010, Vieira et al., 2009).

Manufacture of semi-IPN hydrogels based on MCC from cellulose pulp for biomedical applications. MCC was dissolved in PEG / NaOH solvents with N' crosslink acrylic acid monomers, Nmethylenbisacrylamide by free radical polymerization. Increased crosslink concentration causes a decrease in water absorption. The increasing number of crosslinks will affect the polymer chain, thereby reducing the incoming water and causing the chain to become stiff (Bajpai & Swarnkar, 2014). Manufacturer hydrogels from Polyacrylic acid (PAA) with micro-crystal cellulose amplifier (MCC) carried out through acrylic acid polymerization using UV light. Addition of 1% MCC (1% of the weight of acrylic acid) caused an increase in water absorption capacity in the hydrogel of 122 g water / g hydrogel (from 427 to 549 g water / g hydrogel). The water capacity of the hydrogel matrix network increases because the formed hydrogels have a porous structure and have increased surface area in the hydrogels (Ni, Wen. & Liu. 2015).

The addition of cellulose has many advantages, including from plants with available widely (Anah & Astrini, 2015), cheap, and not toxic. Also, the addition of micro-sized cellulose such as microcrystalline cellulose to the polymer matrix can increase the physical strength of the formed hydrogels (Spagnol et al., 2012). MCC is an available widely commercial material (Brinchi, Cotana, Fortunati, & Kenny, 2013) which can be used as fillers, additives, cosmetics, tablets, and food products (Ioelovich & Leykin, 2006).

Based on the description above the researchers were interested in research on the thermal and morphological properties of a polyvinyl alcohol-based hydrogel containing microcrystalline cellulose (MCC) using N'N-Methylene bisacrylamide as a crosslinking agent and ammonium persulfate as initiator.

#### 2 MATERIALS AND METHODS

#### 2.1 Materials

Commercial Cellulose Microcrystalline (Avicel PH 101 50  $\mu$ m), Acrylic Acid (Merck Schuchardt from Germany), Polyvinyl Alcohol, as initiator has used Ammonium Persulfate ( $\geq$  98% from China), N'N-Methylene bisacrylamide (Sigma Aldrich) was used as a crosslinking agent.

### 2.2 Preparation of Hydrogel

Microcrystalline cellulose was heated with 25 mL aquadest at 60°C and stirred for 30 minutes until homogeneous. Polyvinyl alcohol is heated at 85°C added to microcrystalline cellulose and stirred. 0.06 g Ammonium Persulfate is added to the polymer mixture at a temperature of 60-70°C and stirred for 30 minutes. 4 g of acrylic acid which has been added with 10 mL of aquadest and has been neutralized with NaOH 17.5% until pH = 5 is added to the polymer mixture. 0.2 g of N'N-Methylene bisacrylamide was added to the mixture and stirred. The reaction was finished at 70°C after the reaction has been running for 30 minutes. The product is released in hot conditions. The hydrogel was washed with water then the hydrogel is dried in the oven at 60°C. After the dry hydrogel is inserted into the desiccator. MCC and PVA variations can be seen in Table 1.

Table 1: Variation of MCC and PVA.

| MCC (g) | PVA (g) |
|---------|---------|
| 0.0     | 1.0     |
| 0.2     | 0.8     |
| 0.4     | 0.6     |
| 0.6     | 0.4     |
| 0.8     | 0.2     |

#### 2.3 Swelling Test

Three or four replicas of each dried hydrogel were swollen in deionized water at room temperature for 3 days to achieve equilibrium swelling. The degree of swelling of hydrogels were measured after 5 min, 10 min, 20 min, 0.5 h, 1.5 h, 1 day, 2 days and 3 days. Completely three replicas were measured, the standard deviations were marked with error bars in the swelling profile charts. The degree of swelling was calculated as the following Yang (2012) equation:

Degree of swelling = [(Wet weight – Dry weight) / Dry weight]  $\times 100\%$  (1)

### 2.4 Functional Group Analysis

Detection of functional group, was analyzed by a Perkin Elmer-Fourier transform infrared and transmission was measured in the range of 4000–600 cm<sup>-1</sup>

#### 2.5 Morphological Analysis

Changes in the surface morphology of the hydrogel were analyzed by scanning electron microscopy (SEM). The micrographs of samples were taken using a Hitachi TM3000 scanning electron microscopy (SEM).

#### 2.6 Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) was performed using a DSC-60 Plus Shimadzu containing nitrogen gas and its flow rate was 30 mL/min. Hydrogel samples and  $Al_2O_3$  comparison materials were weighed and heated at room temperature to 600°C at a heating rate of 15°C / minute.

#### **3 RESULTS AND DISCUSSION**

#### 3.1 Preparation of Hydrogel

The manufacture of polyvinyl alcohol-based hydrogels containing microcrystalline cellulose with acrylic acid monomer, N'N-Methylene bisacrylamide as a crosslinking agent and ammonium persulfate as initiator. Polyvinyl alcohol and cellulose microcrystal were varied to produce five hydrogels can be seen in Figure 1.



Figure 1: Hydrogel with varied microcrystal cellulose.

#### 3.2 Swelling Test

Swelling test is done by determining the percent swelling ratio. The percentage of the hydrogel swelling ratio can be seen in Figure 1. Based on Figure 1. It can be seen that at 5 and 10 minutes. Hydrogel 3 has the highest percent swelling. At 20 minutes the hydrogel 5 has the highest percent swelling. The highest swelling percentage at 30 and 90 minutes was shown in hydrogel 1. On the first, second, and third days hydrogel 4 had the highest percent swelling. It can be assumed that the hydrogel 4 has the highest percent swelling of around 3245%.

Water absorption from hydrogel 4 has increased. This is because the addition of the amount of MCC 0.6 g to the hydrogel 4 affects the percent swelling ratio in the hydrogel. The addition of MCC will increase the percent swelling ratio but the addition of 0.8 g MCC percent swelling ratio decreases which occurs in hydrogel 5. Water absorption will decrease too.

The addition of 0.2 g MCC of Hydrogel 2 and 0.4 g MCC of hydrogel 3 had lower percent swelling compared to hydrogel 1 without the addition of MCC.



Figure 2: Degree of swelling (minutes).



Figure 3: Degree of swelling (days).

#### 3.3 Analysis of FTIR

Functional group analysis using FTIR is a qualitative analysis used to interpretation absorption peaks from the infrared spectrum. This analysis can show various areas of hydrogel absorption that are produced so that the resulting functional group changes can be produced. Data analysis of functional groups using FTIR is presented in graphical form in Figure 4 and Table 2.

The FTIR spectrum of hydrogel 1 and hydrogel 4 produced showed several absorption peaks is hydrogel 1 absorption at 3340, 2924, 2181, 1713, 1565, and 1404 cm<sup>-1</sup>, and hydrogel 4 absorbed at 3336, 2945, 2167, 1707, 1558, and 1402 cm<sup>-1</sup>.

The FTIR spectrum of hydrogels 1 and 4 is a wide absorption peak at 3340 and 3336 cm<sup>-1</sup> wavenumbers which shows the presence of vibrating O-H groups from microcrystalline cellulose, polyvinyl alcohol, and acrylic acid so the absorption

appear wide (Sunardi, Irwan, Nurjannah, & Istikowati, 2013). The absorption peak at 2924 and 2945 cm<sup>-1</sup> on hydrogel 1 and hydrogel 4 showed the presence of C-H stretching. Also, there is an absorption peak at wave number 2181 and 2167 cm<sup>-1</sup> which shows CH<sub>2</sub> in crosslinks (Saragih, Tamrin, Marpongahtun, Nasution, & Abdillah, 2018).

The presence of a functional group C = O of acrylic acid at the absorption peak was 1713 and 1707 cm<sup>-1</sup>. Curved absorption sharp peaks at wavenumber 1565, 1404 cm<sup>-1</sup> from hydrogel 1 and absorption peaks at wavenumber 1558 and 1402 cm<sup>-1</sup> from hydrogel 4 showed symmetrical and asymmetrical stretches of carboxylic anions (COO-) (Bajpai & Swarnkar, 2014).

The results of FTIR data obtained can be concluded that there was not significant change in functional groups on hydrogel 1 and hydrogel 4. This is because hydrogels only occur physical interactions on hydrogen bonds between O-H functional groups of MCC, carboxylates, and polyvinyl alcohol.



Figure 4: FTIR spectra of Hydrogel 1 and Hydrogel 4.

Table 2: Functional group of hydrogel.

| Wavelength (cm <sup>-1</sup> ) | Functional Groups |
|--------------------------------|-------------------|
| 3340 and 3336                  | O-H               |
| 2924 and 2945                  | C-H               |
| 2181 and 2167                  | CH2               |
| 1713 and 1707                  | C=O               |
| 1565 and 1558                  | C00-              |
| 1404 and 1402                  | COO-              |

#### **3.4 Differential Scanning Calorimetry**

The DSC of the hydrogel is shown in Figure 5. The DSC thermogram of Hydrogel 1 obtained 4 peaks. At the first peak, there was an endothermic reaction

from a temperature of 162.31°C to a temperature of 177.48°C which required the energy of -111.12 mJ or equivalent to -26.55 mcal. The second peak of Hydrogel 1 has an endothermic reaction from a temperature of 176.21 to a temperature of 190.02°C which requires the energy of -343.73 mJ or equivalent to -82.11 mcal. Hydrogel 1 experienced melt at 167.95°C and the perfect melt occurred at 177.53°C. At the third peak, an exothermic reaction occurs. Hydrogel 1 began to increase in temperature from 253.96°C to a temperature of 308.97°C which released the energy of 260.47 mJ or equivalent to 62.22 mcal. At this peak of hydrogel 1 is degraded at 296°C where the tissue arranged in the hydrogel begins to weaken the bonds with each other. At the fourth peak, an exothermic reaction occurs. Hydrogel 1 experienced an increase in temperature from 445.51°C to 507.40°C which released the energy of 2.94 J or equivalent to 702.34 mcal. At this time the three-dimensional network structure on hydrogel 1 experienced decomposition. It means the composition of hydrogel has burned to be ash and water vapor. We can see the fourth peak in figure 4. Hydrogel 1 has good thermal stability occurs at a temperature of 482.28°C.

The DSC thermogram from Hydrogel 4 obtained 3 peaks. At the first peak, there was an endothermic reaction from a temperature of 60.69°C to a temperature of 223.40°C which required the energy of -1.49 J or equivalent to -356.69 mcal. Hydrogel 4 experienced melt at 134.02°C. The second Hydrogel 4 peak exothermic reaction occurs.



Figure 5: Thermogram of Hydrogel 1 and Hydrogel 4.

Hydrogel 4 began to experience an increase in temperature from 246.24 to 289.79°C which released the energy of 215.50 mJ or equivalent to 51.48 mcal. At this peak of hydrogel 4 are degraded at a temperature of 275.97°C where the tissue composed of hydrogels begins to experience weakening of each

other bonds. At the third peak, an exothermic reaction occurs. Hydrogel 4 experienced an increase in temperature from 443.14 to 535.98°C which released the energy of 3.73 J or equivalent to 890.16 mcal. At this time the three-dimensional network structure on hydrogel 4 has experience decomposition. It means the composition of hydrogel has burned to be ash and water vapor. We can see the third peak in Figure 5. The thermal stability of hydrogel 4 occurred at 473.79°C. From the picture above it can be seen that the thermal stability in hydrogel 1 is greater than the thermal stability of the hydrogel 4. Thermal stability decreases with the addition of 0.6 g MCC.

#### **3.5 Scanning Electron Microscopy**

The results of the surface morphology analysis of Hydrogel 1 without the addition of MCC, Hydrogel 4 with the addition of 0.6 g MCC, and Hydrogel 5 with the addition of 0.8 g MCC. These results provide information relating to the surface of the hydrogel with homogeneity. The surface morphology of hydrogels was analyzed with 500x magnification.

SEM micrographs of the surface of hydrogel 1 without the addition of MCC in Figure 6 show a homogeneous, smooth, flat surface without pores. It can be concluded that PVA has been distributed to all hydrogel 1 networks.

Figure 7 surface of the hydrogel 4 with the addition of 0.6 g MCC shows a slightly coarse surface, and a surface that looks deep and dark so it looks slightly porous. MCC spreads well in hydrogels. Higher absorption occurs due to the irregular shape of the hydrogels and pores (Saragih et al., 2018).



Figure 6: SEM micrographs of Hydrogel 1 without using MCC.



Figure 7: SEM micrograph of Hydrogel 4 using 0.6 g MCC.



Figure 8: SEM micrograph of Hydrogel 4 using 0.8 g MCC.

Figure 8 the surface of hydrogel 5 with the addition of 0.8 g MCC shows the surface of the hydrogel is not homogenous, coarse, and it looks a lot porous. Lots of porous because the shape of the surface looks a lot in the inner and dark areas.

## 4 CONCLUSIONS

From the research that has been done on the thermal and morphological properties of polyvinyl alcoholbased hydrogels containing microcrystalline cellulose, it is undeniably about making optimal mixtures in the manufacture of hydrogels from PVA, MCC and acrylic acid using the ammonium persulfate initiator and crosslinking, N'N-Methylene bisacrylamide by involving (0,4: 0,6: 4: 0,06: 0,2) g. As much as 0.6 g MCC has the highest swelling ratio of 3245% in Hydrogel 4. The higher the swelling ratio, the more optimal absorption. Hydrogels that have absorbency are characterized optimally using FTIR, SEM, and DSC. The results of FTIR data obtained can be concluded that there was not a significant change in functional groups on hydrogel 1 and hydrogel 4. This is because hydrogels only occur physical interactions on hydrogen bonds between O-H functional groups of MCC, carboxylates, and polyvinyl alcohol. Thermal stability decreased with the addition of MCC 0.6 g from a temperature of 482.28°C to a temperature of 473.79°C. It means the addition of 0.6 g MCC can increase absorption and decrease the thermal stability. The Surface morphology of hydrogel 1 looks homogeneous, smooth and has no pore. When the addition with 0.6 g MCC the surface of the hydrogel slightly coarse, and has a slight porous. The porous on the surface of the hydrogel increases when additing of 0.8 g MCC and decreases absorption

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