Characterization of capillary pumping

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Submission date: 15-Apr-2019 05:35PM (UTC+0700)
Submission ID: 1112769712
File name: Characterization_of_Capillary_Pumping.docx (60.67K)
Word count: 4130
Character count: 22335

Characterization of Capillary Pumping in a novel *Sintered* Zeolite and Hybrid Zeolite-Cu for *Wick Heat Pipe* Development

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ABSTRACT

Wick is an important component of the heat pipe. Wick flowing the working fluid from the condenser to the evaporator, driving the working fluid to circulate in the heat pipe so that occurring heat transfer process of heat pipe. To comply these requirements wick must provide wetting surface and capillary effects. In this study, a novel sintered zeolite and hybrid zeolite-Cu implemented as a wick. A quantity important factors were considered and evaluated on sintered zeolite and hybrid zeolite-Cu, such as wick microstructure, capillary pumping performance and wettability. Wick was prepared by sintering method. The granule sizes of zeolite and Cu were 100µm and 200µm. The working fluid that used for testing capillary pumping performance and contact angles was water. Capillary pumping performance tested by electronic balance and recorded by computer. Contact angle is measured by measuring the image of a liquid droplet placed on the wick surface. Capillary pumping performance of zeolite and hybrid zeolite-Cu was examined by comparing with biomaterial wick. The result shows that the best capillary pumping rate reached by hybrid zeolite-Cu 100µm with 25/75% zeolite-Cu composition. The smallest contact angle is obtained by zeolite 200µm. The decrease of CA of hybrid zeolite-Cu would increased the wettability and capillary pumping performance, but this phenomena was occur only in hybrid zeolite-Cu 100µm with 75/25% and 50/50% zeolite-Cu composition, zeolite 200µm and hybrid zeolite-Cu with 75/25% zeolite-Cu composition.

Keywords: wick, capillary pumping, wettability, zeolite.

1. Introduction

Heat pipes, vapor chambers, capillary pumped loops (CPL) are two-phase heat transfer devices. Two-phase heat transfer device has little dimension with the capabilities to transport enormous energy, and small temperature drop more desirable at this time. This two-phase heat transfer devices are very high conductivity devices [1]. The phase changes occur very quickly and effectively, from liquid to vapour in evaporator and from vapour to liquid in condenser [2]. The two-phase heat transfer devices have been widely applied in thermal management as cooling of electronic device [3, 4], spacecraft components, heat pump, air conditioning [5], solar heater [6, 7], light emitting diode (LED) and other two phase thermal control [8]. Nowadays two-phase heat transfer devices has developed various type of heat pipe as though straight heat pipe [9-11], loop heat pipe (LHP) [12-15], miniature LHP [16], ultra-thin heat pipe [17].

Heat pipe basically consists of three components; they are container, working fluid and wick. A lot of research had been undertaken to develop components of heat pipe. One of components which will be developed in this research is wick. Wick is a component functioning to generate capillary pressure to flow working fluid from condenser to evaporator [18]. Wick must provide the capillary pressure to driving working fluid circulation. Capillary pumping performance has important effect on the heat transfer capacity of heat pipe. Studies to investigate the capillary pumping performance has been conducted. For example, Deng [19] and Tang [20] employed an infrared (IR) imaging method to monitor the capillary rise process. Li et al [21] is developing a method capillary pumping amount real time changing curve which is recorded by electronic balance and computer.

The other fundamental parameters in heat pipe is wettability. Wettability greatly affects the capillarity of the porous wick. Capillarity or capillary action is the basic driving force for the wicked heat pipe. Capillary pressure transport the working fluid from the condenser to the evaporator, also able to distribute the working fluid around the evaporator section to any areas where heat is received by the heat pipe. Wetting phenomena and capillarity was fundamental parameters in heat pipe [22, 23]. The wetting phenomena is formed when there is contact between solid and liquid, and liquid solid interface is formed because the liquid replaces the gas film on solid. Wetting phenomena or wettability will influence the critical heat flux (CHF), the bubble contact diameter and the nucleation frequency of a pool-boiling heat transfer. The working fluid that pulling back to the hot end by the capillary force shown that wettability of the wicking material directly reduces the critical heat load [24]. The fundamental parameter to measure surface wettability is contact angle (CA). The CA measurement is important parameter to detect material characteristics, obtained solid-liquid interfacial tension, surface roughness, chemical heterogeneity [25]. Based on the contact angle, surface properties has various range i.e. $0^o < CA < 90^o$ for hydrophilic (surface wetting), $90^o < CA < 180^o$ for hydrophobic (non wetting), $150^o < CA < 180^o$ for superhydrophobicity and almost 0^o for superhydrophilicity.

In pursuance of Hebbar [26], there are several methods for measuring CA, such as sessile drop technique, captive bubble method, Wilhelmy plate method, capillary rise at a vertical plate. Sessile drop technique measure CA from the image capture of the droplet on the flat surface [27-29], usually performed using a goniometer. Goniometer usually composed of three parts such as background light, sample stage and camera. Captive bubble method measure CA by capture the image of contact angle of water droplets or air bubbles with the camera [30, 31]. The method that no need to establish the three phase contact line is called Wilhelmy palte method. This method used the smooth and thin vertical plate such as platinum foil or microscope cover glass, brought into contact with testing liquid [32-34]. The last method is capillary rise at a vertical plate, which tested the CA of the liquid comes in contact with a vertical plate. This method ascertained the capillary rise at that vertical plate [35].

The contact angle can be related to the surface energies of three interfaces or defined as three surface tension that is solid-gas surface tension, solid-liquid surface tension and liquid-gas surface tension [36, 37], the relation is shown in Young's equation and Fig. 1.

$$Cos\ \theta = \frac{\gamma_{s-g} - \gamma_{l-s}}{\gamma_{l-g}} \quad (1)$$

where, $\gamma_{s\text{-}g}$, γ_{l-s} and $\gamma_{l\text{-}g}$ are the solid–gas surface tension, the liquid–solid surface tension and the liquid–gas surface tension respectively; θ refers to contact angle.

At present, the method to measure the contact angle is by dripping liquid on a solid surface, taking the image of the liquid droplet placed on the surface and measuring the image to obtain the contact angle [38].

Currently, wick is made of metal such as copper [39, 40], stainless steel [41], metal alloy such as Cu-Ni [42], Al-Ti-Mg [43]. which is easily oxidized and often caused corrosion problems. The oxidation in the heat pipe caused the loss of hydrophilicity and this condition will affect the heat pipe thermal performance, force capillarity and working fluid boiling process. Research of usage of natural material or non-metal material for wick has been conducted that is biomaterial [44]. The biomaterial Tabulate wick can decrease the thermal has been investigated resistance of the LHP, furthermore improve the LHP performance. Nevertheless, the utilization of biomaterial Tabulate feared will damaging the biomaterial Tabulate life. Other natural material that can be applied as a wick was zeolite.

Among the various natural material, zeolite was considered promising due to their unique properties. Zeolites have some specific properties such as adsorption-desorption capacity, ion exchange capacity, hydrophilicity, high thermal and chemical stability, micro-porosity, non-toxicity and eco-friendly nature [45-48]. Zeolites are non-metallic minerals, and the minerals in zeolite group generally found in tufa rocks formed from volcanic-ash sediments. Zeolites have unique crystal structure like a honeycomb, that can release water by heating. Zeolite consists of crystal structure $(AlO_4)^{5-}$ and $(SiO_4)^{4-}$. Zeolites have been used in various fields such as oil refining, petro chemistry, agriculture, water and waste water treatment. Although has ability as a fluid absorber, zeolite has tendency as a conductor, so will be tried to hybrid zeolite with metal material such as copper in this experiment.

Studies on wick heat pipes using metal and biomaterials have been conducted, but natural rock such as zeolite used as a wick material has never been studied. The purpose of this study was to investigate and characterize the application of zeolite as wick in heat pipe. Wick materials have several requirements such as capillary, permeability and good pore structure, because wick must generate capillary pressure to flow working fluid from condenser to evaporator. Preliminary testing such as porosity and capillary pumping will be required to make zeolite suitable for use as a wick. Porosity, particle size and shape of zeolite sample will measure with SEM and chemical composition will test with Energy Dispersive X-Ray Spectroscopy (EDS). Capillary pumping amount of zeolite will be test by capillary pumping test.

2. Research methodology

2.1 Sample Preparation

In this experiment zeolite was crushed to obtain smaller size. Zeolite powder was sieved into two size range: 100 µm and 200 µm. For the hybrid zeolite-Cu, zeolite with a particle size 100 µm were hybrid with Cu 100 µm and zeolite with a particle size 100 µm were hybrid with Cu 200 µm. Zeolite powder and hybrid zeolite-Cu were inserted into the copper pipe before sintering process. The copper pipe size was 65 mm and 9.6 mm for long and outer diameter respectively. The sintering process regulates temperatures at 950℃ for 60 minutes in a furnace with programmable temperature controller.

Figure 2. shows sample pipe size, sintered powder zeolite and hybrid zeolite-Cu sample for grain sized 100 µm and 200 µm respectively. The micro-morphologies (SEM) of the final sintered zeolite and hybrid zeolite-Cu were shown in Fig. 2 and 3, the pore size distribution studied by using the image analysis method of those samples. The porosity of the zeolite and hybrid zeolite-Cu was shown in Table 1.

2.2. SEM morphologies

SEM test (Scanning Electron Microscope) was carried out to determine the morphology and microstructure of sintered zeolites. SEM observes very small surface structures, which cannot be done by optical microscopes. The type of SEM testing equipment is JSM-6510. The microstructure of the sample will show the distribution of size and shape of the granules from the sample and this is needed to analyze the porosity of the sample. Porosity analysis obtained by processing the image from the SEM test using ImageJ.

Porosity, shape and size distribution of the sample granules will affect the capillary force of the wick heat pipe. Porosity is the ratio of the void's volume to the entire wick's volume. Porosity stated by Putra [44] is a comparison between volume of pore wick (V_{pw}) and volume of porous media (V_{pm}) based on Archimedes' principle, i.e. measured the weight of the wicks in a dry state, then measured the weights of the wicks saturated with water both in air and floated in water. The equation for calculating porosity (ε) is :

$$Porosity\ (\varepsilon) = \frac{V_{pw}}{V_{pm}} \quad (2)$$

The micro-morphologies (SEM) of the final sintered zeolite and hybrid zeolite-Cu were shown in Fig. 3 and Fig. 4, the pore size distribution studied by using the image analysis method of those samples. The porosity of the zeolite and hybrid zeolite-Cu was shown in Table 1.

2.3. Capillary Pumping Performance

The method of measuring the capillary pumping performance of porous wick were used capillary pumping amount real time changing curve method. Capillary pumping amount real time changing curve method has been performed by Li et al. [21], there are three obvious important parameters i.e. capillary pumping amount, capillary pumping time and capillary pumping rate. Capillary pumping amount was a method that performed by measuring the mass of liquid that can be transported in the porous wick. Capillary pumping time shows the time that the porous wick needs to reach saturation from the beginning of the capillary pumping. Capillary pumping rate measuring the rate of porous wick reaches saturation. In this study capillary pumping amount and capillary pumping time were performed to test the capillary pumping of the zeolite and hybrid zeolite-Cu samples.

Fig. 5 shows the schematic diagram of capillary pumping test. The necessary equipment is a breaker placed on an analytical balance. The holder placed beside analytical balance to hold and push down the sample until the bottom sample touched the surface of the working fluid. Working fluid pumped from the breaker to the wick when the wick reaches the surface of the working fluid due to capillarity. The amount of working fluid being pumped is equated to the reading reduction of the electronic balance.

The accuracy of the electronic balance used in this study is 0.1 mg. Capillary measurement is carried out at atmospheric pressure.

2.4. Contact Angle

The equipment to test contact angle consist of a horizontal stage to hold the sample, a syringe to form liquid drop, light for lighting, and High Speed Video Camera (HSVC) to record liquid droplet. The liquid droplets were located on a flat surface of sample. Distilled water was used as liquid to be tested. Destillated water was droped on the surface sample then the contact angle were measured from the images captured by the computer software.

The syringe must remain in the liquid drop during measurement to avoid undesired vibration. The needle diameter should be as small as possible so it does not distort the drop profile shape. Because, the drop might be unsymmetrical, it is advisable that contact angles be measured on both sides of the liquid drop profile, and to use the averaged result. For a relatively large substrate, contact angles should be measured at multiple points to give an average value that is representative of the entire surface. The equipment that used for contact angle measurement is seen in Fig. 6.

To measure the CA, computer software were needed. Water droplets were captured six times on each sampel under the condition 1 atm, 20^{o}C and relative humidity level of 65%, and their average values were used. Six samples with various surfaces were used to measure static contact angles. For each solid surface, the measurements were repeated for six times. Fig. 7 and Fig. 8 shows the corresponding images of drops formed on the surfaces of zeolite and hybrid zeolite-copper. Contact angle measurement were carried out using ImageJ program.

The result of contact angle measurement are used to obtain capillary pressure. The capillary pressure generate liquid rise along the wick, the liquid–vapor interface plays the driving force [19]. To obtain the capillary pressure used Laplace–Young equation as follows:

$$\Delta P_{cap} = \frac{2\sigma cos\theta}{r_p} \quad (3)$$

where σ is the surface tension of liquid, r_p is the pore radius, and θ is the contact angle between solid and liquid.

Effective capillary radius (r_{eff}) usually replaced the pore radius and the contact angle cosine in order to characterize the wicks more simply, thereby yielding :

$$r_{eff} = \frac{r_p}{cos\theta} \quad (4)$$

Thereby, the capillary pressure is :

$$\Delta P_{cap} = \frac{2\sigma}{r_{eff}} \quad (5)$$

To characterize the permeability of a porous wick that the shape of particle is irregular, used the Balke–Kozeny equation, which is known can predict permeability accurately [20]. The equation is :

$$K = \frac{d_p^2 . \varepsilon^2}{150\,(1-\varepsilon)^2} \quad (6)$$

where d_p is the particle diameter, ε is the porosity of wick structure and 150 is a geometrical factor (C) depending on the powder characteristics and fabrication process. For the irregular sintered wicks, the C value is 150 or 180.

3. Result And Discussion

3.1. Capillary pumping amount and capillary pumping time of zeolite and hybrid zeolite-Cu

In this study capillary pumping amount and capillary pumping time was assessed by measuring the mass of liquid that can be transported in the porous wick at given time. Capillary pumping amount was two of the three parameters of the capillary pumping amount real time changing curve method. The other parameters is capillary pumping rate. Capillary pumping time was performed by measuring the time that the zeolite and hybrid zeolite-Cu samples reach saturation. Capillary pumping rate indicates capillary pressure and flow resistance of the zeolite, hybrid zeolite-Cu samples and working fluid, these illustrated by the rapidness or tardiness of the porous wick to reached saturation.

From Fig. 9 shows the capillary pumping amount of zeolite and hybrid zeolite-Cu 100 µm grain size. The composition of 25/75% zeolite-Cu has the best capillary pumping amount than the other three compositions. 25/75% zeolite-Cu was the best can reach 1.5 gram saturation in 20 second. The 50/50% zeolite-Cu composition reach 1 gram saturation in 10 second. Nevertheless, two other composition only reach saturation below 1 gram saturation. The reason is zeolite were hydrophilic due to molecular pore size. The molecular pore size made zeolite function as molecular sieve, zeolite has the ability to adsorb water up to 22% by weight [49, 50]. And on the hybrid, copper granules were coated with zeolite so that the zeolite will form groove on copper grains which increases the ability of wick capillarity.

Fig. 10 shows capillary pumping amount of zeolite and hybrid zeolite-Cu 200 µm grain size. In this size, 100% zeolite composition was the best capillary pumping amount. The next good capillary pumping amount were composition of 25/75% zeolite-Cu, 75/25% zeolite-Cu and 50/50% zeolite-Cu. Composition of 100% zeolite reach 1 gram saturation in 20 second. Composition of 25/75% zeolite-Cu reach 0.8 saturation in 50 second. Composition of 75/25% zeolite-Cu reach 0.7 saturation µm in 35 second, and the last was composition of 50/50% zeolite-Cu, reach 0.35 gram capillary pumping amount in 15 second.

From the Fig. 9 and Fig. 10 states the best capillary pumping amount were different in two types of material grain size. The best capillary pumping amount for 100 µm grain size was 25/75% zeolite-Cu and the best capillary pumping amount for 200 µm grain size was 100% zeolite. The 100 µm grain size obtain higher capillary pumping amount than 200 µm grain size. As stated by Li et al. [21], the porous wicks with tighter granules and straighter channels in the microstructures were have better capillary pumping performance. Grain size influence the porosity, the increase in grain size porosity will declines. Declined the porosity of zeolite 200 µm grain size will inhibit capillary pumping time.

Comparing the capillary pumping performance between zeolites, hybrid zeolite-Cu, biomaterial and Cu was carried out by comparing of capillary pumping amount, capillary pumping time and capillary pumping rate. From the capillary pumping time parameters, seen that the capillary pumping time of zeolite and hybrid zeolite-Cu is about 20 seconds, equal to capillary pumping time of biomaterial and Cu was about 10 to 20 second [44]. The capillary pumping time of biomaterial and sintered cooper was about 10 to 20 second, stated in Fig. 11. Although capillary pumping amount zeolite and hybrid zeolite-Cu lower than biomaterial, but still close to Cu.

3.2. Effect of porosity and microstructure on capillary pumping amount

Capillary pumping performance of samples greatly influenced by porosity and microstructure of the samples. From Fig. 12 and Fig. 13 the mean pore diameter of 100 µm grain sized were 1.6, 1.74, 1.83 and 1.73 for 100% zeolite, 75/25% zeolite-Cu, 50/50% zeolite-Cu and 25/75% zeolite-Cu respectively. The mean pore diameters of 200 µm grain sized were 3.18, 2.26, 2.63 and 3.08 for 100% zeolite, 75/25% zeolite-Cu, 50/50% zeolite-Cu and 25/75% zeolite-Cu respectively. Besides the mean pore diameter, the distribution of pore size also affects porosity. Pore diameter distribution of zeolite and hybrid zeolite-Cu grain sized 100 µm and 200 µm respectively was dominated in 1 µm. The amount of 25/75% zeolite-Cu, 100 µm grain size was 40.000. The amount of 100%

zeolite, 200 µm grain size was 4300, less than 100 µm grain. This causes 100 µm grain size samples obtain better capillary pumping.

Pore radius diameter affect the porosity of the porous wick. Based on Table 1, the porosity of 100 µm grain sized were 12.96%, 15.01%, 20.68% and 22.36%, greater porosity than 200 µm grain sized i.e. 4.95%, 2.91%, 2.48% and 4.51% for 100% zeolite, 75/25% zeolite-Cu, 50/50% zeolite-Cu and 25/75% zeolite-Cu respectively.

In hybrid zeolite-Cu porosity obtained because Cu has a lower melting point than zeolite, when sintered with temperature close to zeolite melting point it will serve as a binder between zeolite grains. Copper will form grooves on the surface of zeolite grains. Grooves in zeolite granules will increase capillary pumping amount wick.

Zeolite and hybrid zeolite-Cu with 100 µm grain size has homogen pore size and more pore than zeolite and hybrid zeolite-Cu with 200 µm grain size. So that zeolite and hybrid zeolite-Cu with its smaller grain, homogeneous grain and microporous properties cause wick sample has better capillary pumping performance. Capillary pumping performance was increased with the increasing porosities of the porous wicks.

In hybrid zeolite-Cu, zeolite particle protects metal particles from corrosion, and the Cu particle plastically deforming to improve a crack propagating through the zeolite matrix of the hybrid zeolite-Cu[51].

3.2. Contact Angle Analysis

The recorded drop images were analyzed by ImageJ and the contact angle value are plotted in Fig. 14. Fig. 14 shows that six samples tested resulting in contact angles smaller than 90°. Due to microporous properties of zeolites [45] resulting contact angle smaller than 90°. Exhibited in 100% zeolite composition, result contact angle smaller than hybrid zeolite-copper. The contact angle larger in the hybrid composition of 75%zeolite-25%Cu, decreases in the hybrid composition of 50%zeolite-50%Cu and increases again in the composition of 25%zeolite-75%Cu.

To examine the surface roughness effect on the contact angle value, six repeated contact angle measurements were performed on each sample. Surface roughness is indicated by particle size. The surface more rough as the particle size larger. The surface sample of 200µm grain particle is more roughness than the sample with 100 µm grain particle. Fig. 15 shows the contact angle in the samples with particle size of 100µm is greater than the contact angle in the samples with particle size 200µm, the contact angle value decreases with the increase of surface roughness. It was concluded that the solid surface with the bigger surface roughness has the smaller contact angle which is also stated by Ngo & Chun [52]. The wetting surface is enhanced by roughness is inherent with the Wenzel state i.e., increase in surface roughness will make a hydrophilic surface more hydrophilic and a hydrophobic surface more hydrophobic [53].

The decrease of CA will increase the wettability and the capillary force. The capillary force cause the liquid transport. Liquid can be transported by tension forces, which are caused by capillary force. In heat pipe good capillarity indicates how well the liquid move from condenser to evaporator. Capillarity forms the main criteria in evaluating heat pipe performance. Good capillarity will cause good heat transfer to obtain an efficient heat transfer device.

4. Conclusions

In this study a novel sintered zeolite and hybrid zeolite-Cu was assessed using capillary pumping amount. Capillary pumping amount was measuring the mass of liquid that can be transported in the porous wick at given time. Furthermore, zeolite and hybrid zeolite-Cu sample was tested by micro-morphologies (SEM) and the pore size distribution studied by using the image analysis method. The capillary pumping amount of zeolite and hybrid zeolite-Cu exhibit that zeolite and hybrid zeolite-Cu as an alternative wick. The best capillary pumping amount was reached by hybrid zeolite-Cu 100 µm grain size with a composition of 25/75% zeolite-Cu. Hybrid zeolite-Cu with its smaller grain, homogeneous grain and microporous properties cause wick sample has better capillary pumping amount. The capillary pumping time of hybrid zeolite-Cu 100 µm grain size equal to the capillary pumping time of biomaterials for about 20 second.

This study developed sintered zeolite wick and hybrid zeolite-copper wick, that introduces a natural wick and sintering process to fabricate the wick. To fulfil the fundamental parameters in heat pipe, the zeolite wick and hybrid zeolite-copper tested the wettability and capillarity performance. The wettability and capillarity in wicks was characterized using an CA measurement method. The test liquids used DI water and the droplet image capture were process with computer software to obtain the contact angle. The main conclusions can be summarized as follows:

1. The Contact angle (CA) decrease with an increase surface roughness of the sample wick. The surface sample of 200µm is more roughness than the sample with 100 µm grain particle.
2. The Contact angle (CA) decrease, wetting phenomena at the surface sample wick increase, and the capillary pumping force will improved.

The water can be transported by capillary force. The capillarity increases when CA decreas, and surface roughness increases. The optimal wick should be in addition to the surface properties.

Acknowlegement

The authors would like to acknowledge Udayana University and Applied Heat Transfer Research Group University of Indonesia for laboratory facilities. The authors greatly appreciate the support and helpful suggestions.

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