DIVISION OF PARAFFIN MELTING ZONE BASED ON MULTISCALE EXPERIMENTS

by

Xiaoqing LI[a,b], Renqiang LIU[a,c], Tianyu ZHANG[b], Peng YU[b*], and Xiaoyan LIU[b*]

[a] Changshu Institute of Technology, Changshu, China
[b] School of Civil and Engineering, Northeast Petroleum University, Daqing, China
[c] School of Petroleum Engineering, Northeast Petroleum University, Daqing, China

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Phase change energy storage materials are widely used in the field of renewable energy. Paraffin is one of the common phase change energy storage materials. As a multi-component hydrocarbon mixture, the melting of paraffin is different from that of pure substance. In addition the solid and liquid zones, there is also a fuzzy zone in which solid and liquid coexist. In this paper, the melting characteristics of paraffin in phase transition zone are studied by multi-scale experiments. Through the visualization experiment of square cavity paraffin melting, the solid zone, fuzzy zone and liquid zone are determined, and the moving process of phase interface is tracked by digital pictures and infrared heat maps. The evolution process of the pore structure in the fuzzy zone under different temperatures is photographed by means of the micro-experiment, and it is revealed that there are two areas in the fuzzy zone, porous media area and multi-phase flow area. The results show that the melting process of paraffin can be divided into four zones: liquid zone, multi-phase flow zone, porous media zone, and solid phase zone. According to the polarizing optical microscopy picture, the continuous phase and discrete phase transition relationship between solid wax crystal and liquid paraffin is captured. The polarizing optical microscopy picture is statistically analyzed, and the critical liquid phase ratio of the transition from porous media area to multi-phase flow area is given under experimental conditions.

Key words: *phase change, melting, multiscale experiments, fuzzy zone*

Introduction

Phase change heat transfer means that heat transfer occurs when phase change occurs, and heat storage or release is realized. Phase change heat transfer technology has been widely used in engineering. Gas-liquid phase variation, such as condensers in steam turbines, water cooling walls in boilers, condensers and evaporators in refrigeration (or heat pump) devices, *etc.* Solid-liquid phase transformation, such as air-conditioning cooling technology, waste heat utilization technology, solar energy utilization technology, building energy-saving equipment, *etc.* In various engineering fields, PCM are especially widely used in the field of solar energy [1-5]. The extensive application of phase change heat transfer in various engineering fields is inseparable from the scholars' continuous research on PCM. Paraffin is a common PCM. When paraffin as a PCM melts and absorbs heat, the material will have different states at different po-

* Corresponding authors, e-mail: 13945530586@163.com, liu_xydq@163.com

sitions, which are, respectively in liquid phase zone, fuzzy zone, and solid phase zone. Different zones are calculated using different models. The fuzzy zone is generally regarded as porous medium, and the enthalpy-porous medium model is usually used to calculate the fuzzy zone. The work of this paper is to subdivide the fuzzy zone into porous media zone and multi-phase flow zone, so that more accurate models can be selected for different zones and the calculation accuracy can be improved.

The properties of composite PCM are better than those of pure PCM. In the field of composite PCM, the combination of paraffin and foam metal has been studied extensively. In order to study the melting properties of porous media, some scholars have carried out related researches, mainly using visualization experiments. Zheng *et al.* [6] conducted visualization experiments to study the influence of heating position on the thermal properties of foamed Cu-paraffin composite PCM. Jin *et al.* [7] conducted visualization research based on pore size to explore the influence of pore size on melting heat transfer of saturated paraffin in copper foam. The pore size of the melting process was visualized by using the infrared thermal imager, and the transient evolution of the pore size temperature field at typical moments of the smelting process was observed by using the infrared thermal imager. Yao *et al.* [8] conducted visual experimental research on the melt phase transition of paraffin in high porosity open-cell copper foam of pore size. With the help of high definition camera and infrared thermography, the phase-field and temperature field of paraffin foam and pure paraffin were collected, and the melting process was observed intuitively. Hu *et al.* [9] conducted visual experimental research on the heat transfer characteristics of PCM in foam metal with low porosity. The temperature field and solid-liquid melting process of PCM with and without foam were tracked experimentally. The melting time of pure paraffin wax and composite PCM was compared.

Seddegh *et al.* [10] and Gao *et al.* [11], respectively studied the melting process of PCM in vertical cylinders through visualization experiments. The effect of natural-convection on the heat transfer characteristics of PCM was studied by temperature regulation. Mahdi *et al.* [12] took industrial paraffin as PCM and studied the melting behavior of paraffin in different tubular and shell latent heat accumulators through visualization experiments. Reyes *et al.* [13] conducted visualization experiments with emulsions with different volume fractions of paraffin to study the feasibility of replacing part of paraffin with water as PCM.

Through the aforementioned researches, the visualization experiment can intuitively study the melting process from the macroscopic scale and pore scale. Experiments can be carried out in containers of different shapes according to needs. The experimental medium may also be prepared according to the research requirements. Research methods mainly use high definition cameras and infrared thermography to track melting. In this paper, the melting process of paraffin wax is studied by a visualization experiment in a square cavity.

As for the research methods of PCM, some scholars have also carried out researches on the microscopic level of PCM, which cannot only obtain the micro-structure but also observe the evolution process. The SEM and polarizing optical microscopy (POM) are commonly used in the microscopic study of PCM. Li *et al.* [14] prepared porous TiO_2 foams (PTF) by particle-stabilized emulsion method, covered the surface of porous media with carbon layer, and impregnated with paraffin with a certain proportion of solid. The SEM images showed that the prepared PTF had 3-D interpenetrating structures with good porosities. Cheng *et al.* [15] prepared paraffin expanded graphite (EG) composite by vacuum impregnation method. The SEM analysis showed that the paraffin was completely absorbed into the porous network. Wang *et al.* [16] made a microencapsulated composite phase change composites (PCC) with carbon network, and proposed an effective theoretical model to predict the thermal conductivity of composite PCM with different mass fractions of expanded graphite. The PCC of carbon mesh structure was analyzed by SEM. Sari *et al.* [17] studied the transition from crystalline phase to amorphous phase of PCM by using POM, and carried out morphological analysis. Fang *et al.* [18] used POM to study the morphology of microencapsulated PCM. In this paper, POM was used to study the evolution of paraffin melting.

It can be found that macroscopic visualization experiments and microscopic observation are important research methods of PCM. The main content of the research is around the melting process, the preparation of materials and the physical properties of materials. This paper studies the division of melting zone. Solid-liquid phase transformation in phase change heat transfer can be divided into solid-liquid phase transformation of pure substance and solid-liquid phase transformation of multi-component mixture. A pure substance has a fixed phase change temperature, while a mixture has no fixed phase change temperature. The phase change can occur within a certain temperature range, which is called the phase change range. Substances in the phase transition interval are in the state of solid-liquid coexistence, which is called by many scholars as fuzzy or mushy zone. Substances in this interval show different characteristics from solid and liquid and are often regarded as porous media.

Some scholars have studied the solid-liquid interface and the mushy zone. Ding *et al.* [19] conducted an experimental study on the heat transfer characteristics of the phase transition of the alloy, established a dimensionless correlation formula to predict the boundary motion of liquid and solid phase lines. Zhang *et al.* [20] carried out experiments and numerical simulations on the melting heat transfer characteristics of Cu-paraffin foam composite PCM carried out visualization experiments on solid-liquid interface evolution. Yang *et al.* [21] developed an image-based LBM to simulate fluid-flow and predicted the relevant permeability in the fuzzy zone that evolved continuously during the melting and solidification of paraffin. Yang *et al.* [22] also carried out microscopic experiments, and found that the micro-structure evolution of the mushy zone during the melting process was completely different from that during the solidification process, and the melting and solidification of paraffin were not a simple inverse process. Hong *et al.* [23] used the enthalpy porosity method to study the effects of mushy zone constant and gravity acceleration on the thermal properties of PCM during solid-liquid phase change heat transfer.

In order to reveal the melting law within the mixture constraint boundary, capture the moving phase interface and determine the range of the fuzzy zone, a mixture melting visualization experiment in the differentially heated square cavity was carried out in this paper. The existence of fuzzy zone was determined. Then a microscopic experiment of paraffin melting was carried out with the aim of more finely dividing the fuzzy zone. By means of macroscopic visualization experiment and microscopic observation experiment, the division of paraffin melting process was obtained and the method of subdivision of fuzzy zone was given.

Visualization experiment of paraffin melting in square cavity

Design of visual experimental device

Paraffin was extracted from Daqing Oilfield for visualization experiment. The density, thermal conductivity and specific heat of paraffin wax were measured by Hot Disk TPS2200 thermal constant analyzer made in Sweden. The DSC curves were obtained by TA Q200 differential scanning calorimeter made in USA to study the latent heat of phase change. The specific parameters were shown in tab. 1. The experimental device was shown in fig. 1.

Table 1. Physical parameters of paraffin

Physical parameters	Value
Density (solid phase)	880.92 kg/m^3
Density (liquid phase)	775.09 kg/m^3
Conductivity (solid phase)	0.25 W/mK
Conductivity (liquid phase)	0.15 W/mK
Specific heat (solid phase)	2.80 kJ/kgK
Specific heat (liquid phase)	2.25 kJ/kgK
Kinematic viscosity	$3.20 \cdot 10^{-6}$ m^2/s

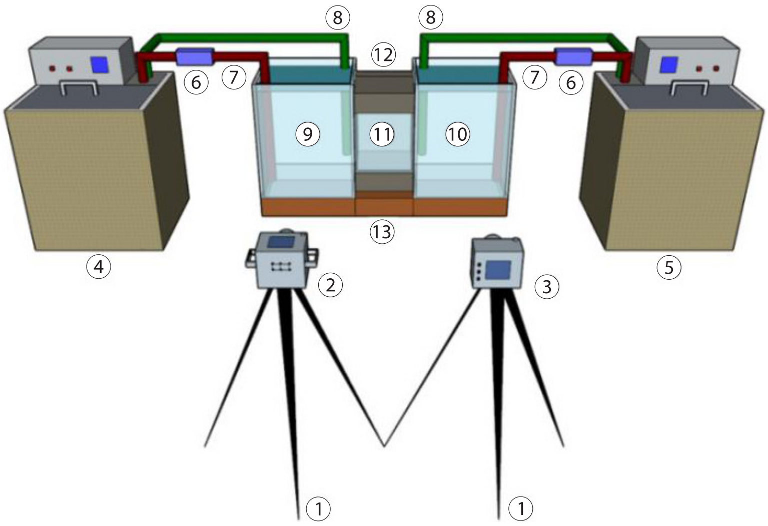

Figure 1. Diagram of visualization experiment equipment for melting in square cavity: *1 – tripod, 2 – infrared thermal imager, 3 – digital cameras, 4 – thermostatic water bath (high temperature) 5 – constant temperature eater bath (low temperature), 6 – circulating pump, 7 – return water pipe, 8 – water supply pipe, 9 – square cavity of constant temperature water (high temperature), 10 – square cavity of constant temperature water (low temperature), 11 – square-cavity paraffin, 12 – thermal isulation materials, and 13 – base*

The experimental apparatus consists of three acrylic chambers, thermostatic water bath system, circulating pump, digital camera, and infrared thermal imager. Square cavity – 11 contains phase change medium paraffin, the square cavity of high and low temperature water bath are – 9, 10, respectively. They are connected to the constant temperature water bath by circulating pump to ensure the constant temperature condition of the left and right walls of the square cavity of paraffin, infrared thermography and digital camera are – 2, 3, respectively, which are used to shoot digital and infrared thermography images of visual experiments. A 5 cm × 5 cm transparent cavity is made with acrylic plate and filled with paraffin. The upper, lower and rear surfaces are wrapped with insulation materials of certain thickness to achieve an approximate adiabatic boundary. The front surface is used to photograph the interface. Constant temperature boundary conditions are simulated by constant temperature water bath at both left and right boundaries. The HH-601 constant temperature water bath is adopted with an accuracy of 0.5 °C. The melting process of paraffin in the square cavity is photographed by Nikon D7000 digital camera and FLIR-T660 infrared thermal imager.

Square cavity of constant temperature water is stabilized at the specified temperature by running two constant temperature water bath devices first. Square-cavity paraffin is placed after temperature stabilization of constant temperature water bath. Every minute, an infrared thermal imager and a digital camera capture the images simultaneously. The solid-liquid interface no longer changes and enters the final state.

Visualization experimental results

Before the experiment, the paraffin were all solid. After the experiment began, with the increase of heating time, the paraffin gradually melted and formed solid/liquid/fuzzy zone in the square cavity. Figure 2 shows the images obtained in the visualization experiment at different moments of the paraffin melting process in the square cavity at the heating temperature of 95.0 °C. At the beginning of heating, the paraffin wax near the thermal boundary melts quickly, but the fuzzy zone is not evident. At the time of 20 minutes, a clear fuzzy zone could be seen. The shape of the fuzzy zone is a *streamer band* with upper narrow width and lower wide width. The melting rate in the middle stage is faster. As time goes by, there are more and more fluids. The hot fluid rises upward due to the buoyancy force, making the top wider and wider. Later stage of the experiment, after 110 minutes, the melting rate obviously slows down, and the phase interface only changes slowly. After 140 minutes, the position of the phase interface and the shape and width of the fuzzy zone remain basically unchanged, and then it is considered that the steady-state is reached.

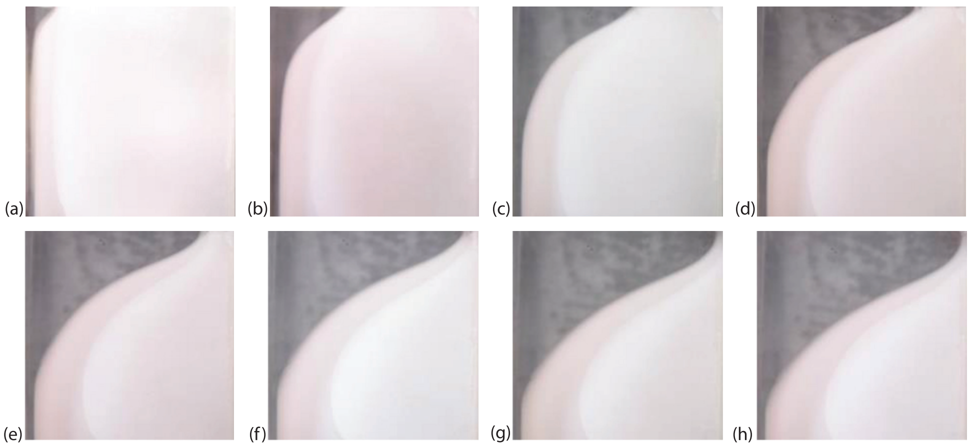

Figure 2. Paraffin melting visualization experiment in square cavity (95 °C); (a) 20 minutes, (b) 40 minutes, (c) 50 minutes, (d) 70 minutes, (e) 90 minutes, (f) 110 minutes, (g) 130 minutes, and (h) 140 minutes

Figure 3 is the infrared thermal image of the melting process taken by FLIR-T660 infrared thermal imager at the heating temperature of 95 °C. Different from the previous ones, the infrared thermal image not only reflects the position of the solid-liquid interface but also reflects the temperature gradient in each region. Uniform color in the liquid phase indicates uniform temperature distribution in the liquid phase. The color changes slightly in the blur area. The color change in the solid phase is obvious and the temperature gradient is large. The different temperature gradient indicates the difference in heating transfer performance.

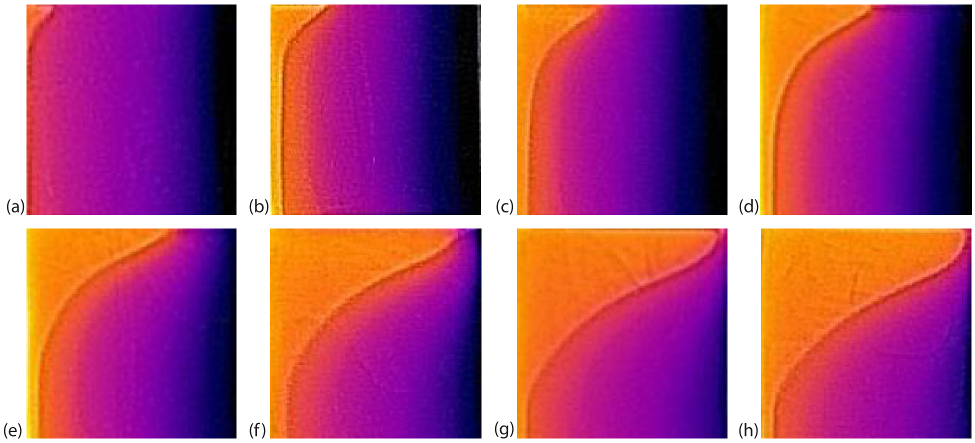

Figure 3. Infrared thermal image of square cavity paraffin melting visualization experiment; (a) 20 minutes, (b) 40minutes, (c) 50 minutes, (d) 70 minutes, (e) 90 minutes (f) 110minutes, (g) 130 minutes, and (h) 140 minutes

As can be seen from fig. 2. The melted paraffin in the square cavity has two clear moving phase interfaces, the liquid phase on the left and the solid phase on the right. The boundary between the two-phases is the liquid-solid mixing zone, also known as the fuzzy zone. Combined with the infrared thermal image taken by the infrared thermal imager and the visual experimental images can determine the approximate boundary position of the fuzzy zone, the solid phase zone and the liquid phase zone, as shown in fig. 4. However, the liquid phase rate change in the fuzzy zone and the specific state of the coexistence of wax crystals and liquid paraffin in the fuzzy zone, namely the pore structure in the fuzzy zone, cannot be obtained from the figure. Therefore, it is necessary to carry out microscopic experiments for in-depth study.

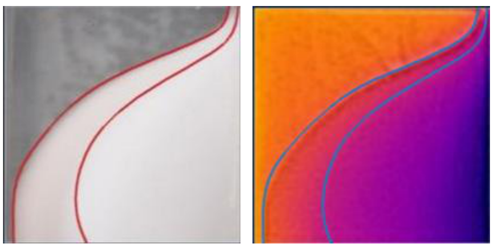

Figure 4. Paraffin melting zone interface of square cavity

Liquid phase rate evolution experiment in the fuzzy zone on micro scale

The existence of the fuzzy zone in the paraffin phase transition process has been proved by visualization experiments, and the temporal and spatial evolution of the melting process has been recorded by digital images, but the specific micro-structure of the fuzzy zone is still unclear. Some scholars indistinct classified the porous media zone and the multi-phase flow zone. But from the digital pictures of the visualization experiment, the overall fluidity of the mushy zone is poor, so the specific structure of the porous media zone and whether there is a multi-phase flow zone are still uncertain. In this paper, the specific structure of the fuzzy zone is determined by microphotography, the flow characteristics are analyzed, the melting zone is divided, and the functional relationship between temperature and liquid rate is determined.

Experimental equipments and methods for liquid phase rate evolution

Paraffin was extracted from Daqing Oilfield for experiment. Polarizing microscope was used to photograph polarizing microimages of paraffin at different temperatures in the phase change temperature range, and then the ratio of wax crystal area was calculated by image processing software, and the liquid phase rate at this temperature was analyzed.

Solid paraffin was placed on the glass slide, and then the glass slide was heated by a constant temperature heating table to melt the paraffin. The melting process was photographed by a microscope.

Experimental results of liquid phase rate

Figure 5 shows the POM pictures of paraffin at different temperatures. The binary algorithm is more applicable and has more physical significance. The POM picture is binarized and the area ratio is obtained by PHOTOSHOP and IMAGE software in order to observe the pore structure more clearly. The liquid-phase ratio in the 2-D plane is approximately calculated, as shown in fig. 6.

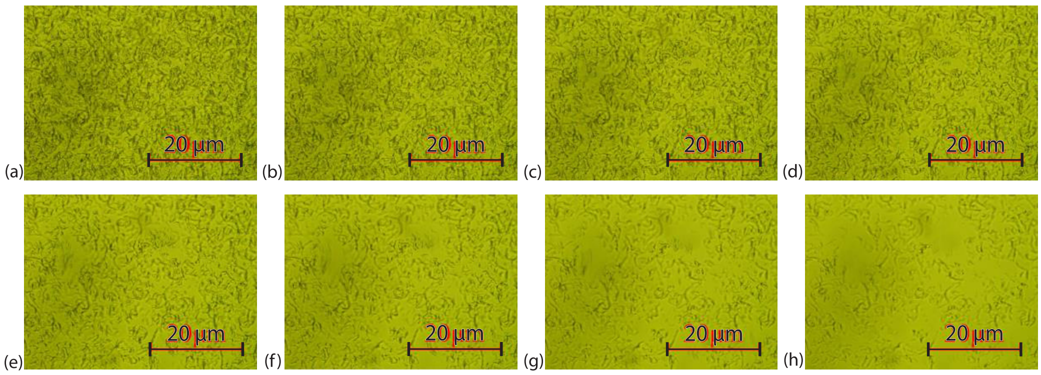

Figure 5. Micrograph of paraffin melting process; (a) 52 °C, (b) 54 °C, (c) 56 °C, (d) 58 °C, (e) 60 °C, (f) 62 °C, (g) 64 °C, and (h) 66 °C

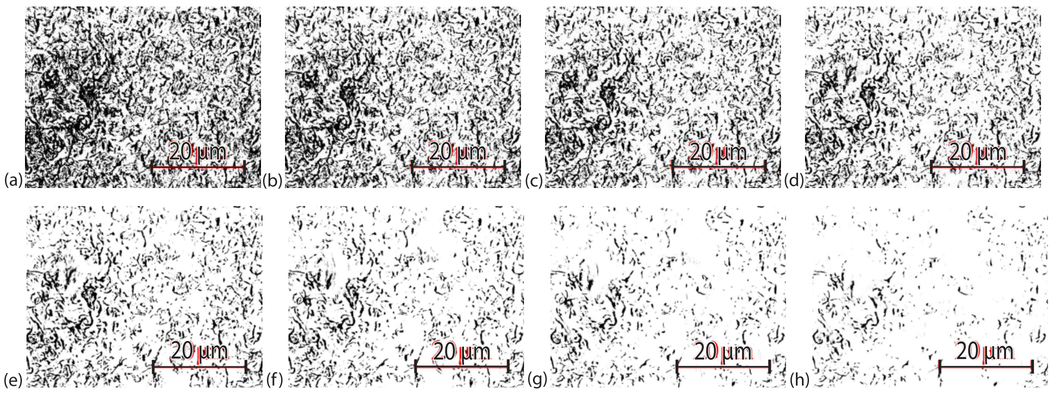

Figure 6. Binary micrograph of paraffin melting process; (a) 52 °C liquid fraction (LF) = 65.74%, (b) 54 °C LF = 72.19%, c) 56 °C LF = 77.43%, (d) 58 °C LF = 81.51%, (e) 60 °C LF = 84.94%, (f) 62 °C LF = 89.21%, (g) 64 °C LF = 93.03%, and (h) 66 °C liquid fraction = 95.95%

As can be seen from figs. 5(a)-5(c), in the initial stage of melting, there are a large number of wax crystals. Solid wax crystals are the skeleton of porous media, and the melted liquid paraffin exists in the pore structure. At this time, it has obvious characteristics of porous media. With the increase of temperature, the solid wax crystal decreases and the porous medium skeleton disappears. As can be seen from figs. 5(f)-5(h), after the temperature continues to rise, a large number of wax crystals melt and disappear, leaving a small number of solid wax crystals suspended in a large amount of liquid paraffin. Liquid phase paraffin is continuous phase, and the solid wax crystal is discrete phase. Figures 5(d) and 5(e) are in the transition stage from porous medium to multi-phase flow.

It can be seen from fig. 6 that the transition from porous media to multi-phase flow zone is not instantaneous, but there is a transition phase, which is difficult to be defined by a value of critical liquid holding rate. Because the liquid phase rate in the fuzzy zone is constantly changing, there is no accurate boundary surface, but in numerical simulation, a critical liquid phase rate is often needed as the boundary between the multi-phase flow region and the porous medium region. It can be seen in figs. 6(d) and 6(e) that with the increase of temperature, the porous media skeleton gradually melts and the wax crystal gradually changes from continuous phase to discrete phase. In order to obtain the evolution law of porous media skeleton structure with temperature during temperature rise. The relevant information about wax crystal particles was analyzed and characterized. Figures 7-10, respectively show the evolution rule of fractal dimension, particle number, average particle area, and average particle diameter with temperature. The wax crystal image captured by polarized light microscope is binarized, and then the binary wax crystal image is lattice processed by MATLAB software. According to the box-counting method, the box dimension of the processed wax crystal lattice is calculated by self programming. The change of box dimension with temperature is counted.

As can be seen from fig. 7, when the temperature is above 60 °C, the fractal dimension gradually deviates from the original linear law. As showing in fig. 8. The number of wax crystal particles decreases slowly within the range of 58-60 °C, because the wax crystals that are stacked to form a skeleton begin to separate at this time. In fig. 9, The average particle area decreased sharply in the range of 58-60 °C. At this time, the gradually separated wax crystals are surrounded by liquid paraffin, and the melting speed is accelerated. In fig. 10, the average particle size also shows a sharp downward trend in the range of 58-60 °C, and the decrease of average particle area of wax crystal leads to the decrease of average particle size. Therefore, it can be considered that the transition from porous media to multi-phase flow zone occurs in the

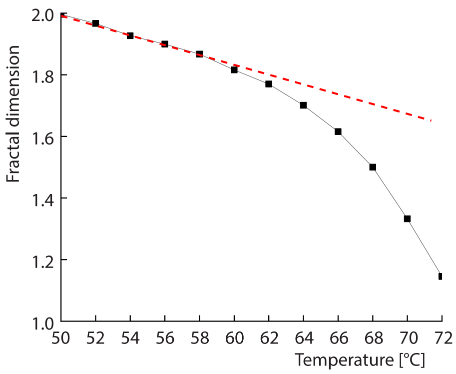

Figure 7. Evolution of fractal dimension with temperature

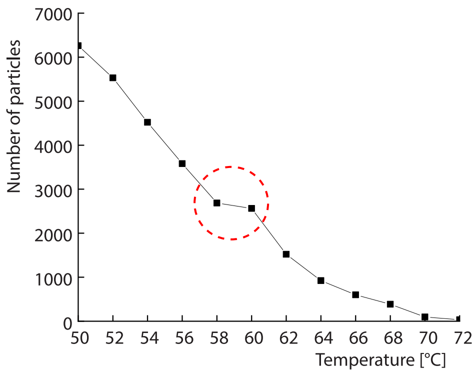

Figure 8. Evolution of particle number with temperature

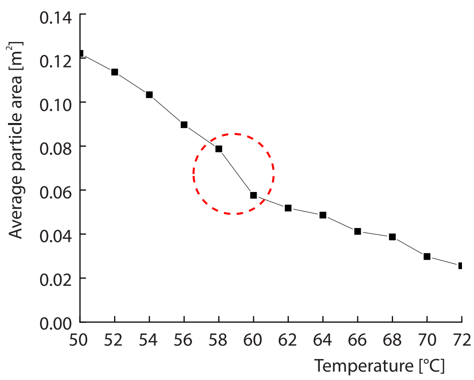

Figure 9. Evolution of average particle area with temperature

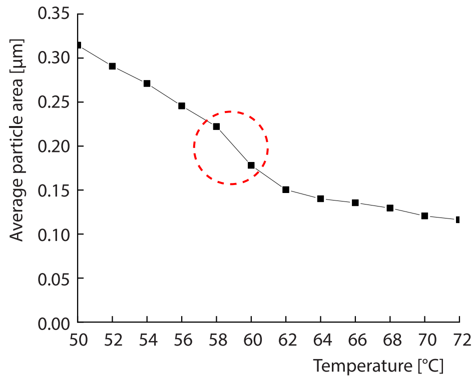

Figure 10. Evolution of average particle diameter temperature

temperature range of 58-60 °C. In this paper, the mean value of the corresponding liquid holdup in figs. 6(d) and 6(e) is 83.23% as the critical liquid holdup for the transition between the porous medium region and the multi-phase flow region divide the melting zone of the mixture.

Regional division of paraffin melting

In the study of solid-liquid phase variation, many scholars adopted REV scale and described its characteristics with apparent liquid holding rate or porosity, not pursuing specific structure of pore scale. The study in this paper also made the same treatment. It has been shown that there is a fuzzy area for the multi-component organic phase transition mixtures such as crude-oil and paraffin.

According to the experimental results, the fuzzy zone can be further divided into two areas: in the area close to the liquid phase, the solid particles are suspended in the liquid phase and flow with the liquid phase under the action of natural-convection. In the zone near the solid surface, the solid wax crystals adhere to the surface, while the liquid paraffin flows between the pores, showing porous media seepage. The liquid phase rate is denoted by the symbol γ. The critical liquid velocity, γ_{tr}, determined by microscopic experiments is used as the boundary between porous media and multi-phase flow zone. In the low liquid phase zone ($0 < \gamma < \gamma_{tr}$), the solid phase is continuous, the liquid phase is discrete, and the flow form mainly considers the porous media seepage. The Brinkmann-Forchheimer-Darcy model is adopted. In the high liquid phase zone ($1 > \gamma \geq \gamma_{tr}$), the liquid phase is continuous and the solid phase is discrete. The multi-phase flow model is adopted. The macroscopic transport equation of pure fluid is directly applied to the multi-phase flow zone. The related physical parameters are characterized by multi-phase flow of composite liquid phase and solid phase.

In the phase transition process, the flow phenomenon exists in the liquid phase zone and the fuzzy zone. Considering the influence of natural-convection in the flow zone, it is assumed that the Boussinesq hypothesis is approximately valid. The fluid is regarded as an incompressible Newtonian fluid, and the volume change of PCM is ignored in the phase transition process.

At this point, the melting zone of the mixture melting process can be divided into four zones, including the liquid phase zone, the multi-phase flow zone, the porous medium zone and the solid phase zone according to the order of the liquid phase rate from high to low. The liquid phase zone is calculated according to the natural-convection heat transfer, using the standard N-S equation. The multi-phase flow zone is calculated according to liquid-solid two-phase flow.

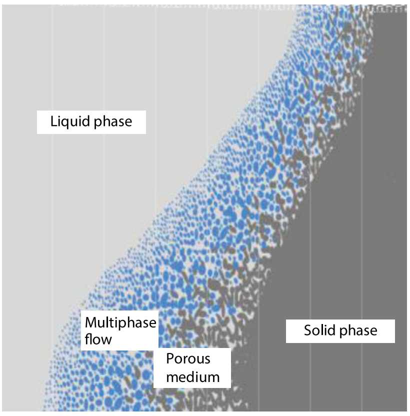

Figure 11. Construction of multi-component material melting zone

Porous media zone is calculated according to porous media seepage. Brinkmann-forchheimer-darcy model was adopted. The heat transfer in the solid phase is pure heat conduction, see fig. 11.

Conclusions

In this paper, the regional division of the melting process of paraffin was studied by experimental means.

- The visualization experiment of paraffin melting in square cavity was conducted, and the thermal temperature boundary was 95 °C. The process of solid-liquid phase transformation was photographed by digital camera and infrared thermography. It entered steady-state at 140 minutes. The existence of solid phase zone, liquid phase zone, and fuzzy zone in the melting process of paraffin wax has been proved macroscopically through experiments. The movement of phase interface during the melting process has been tracked.
- The evolutionary process of the fuzzy zone was photographed by microscope, revealing the existence of porous media and multi-phase flow in the fuzzy zone. The continuous phase and discrete phase between solid waxes and liquid waxes could be analyzed according to POM images. The critical liquid phase rate was defined by statistical method, and the method of dividing the transition from porous medium to multi-phase flow was given.
- The whole melting process was divided based on the critical liquid rate obtained from the two-scale experimental study. The results show that the melting process of paraffin consists of liquid phase zone, multi-phase flow zone, porous medium zone and solid phase zone in the order of liquid phase rate from high to low. An experimental method based on statistical principle is proposed to determine the critical liquid rate in this paper. Using the critical liquid rate to divide the multi-phase flow zone and the porous medium zone can make the numerical simulation more accurate. This study can provide theoretical guidance for the selection of critical liquid rate in numerical simulation.

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