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Fabrication of paraffin wax particles with diameter 100 um --Independent study report

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Abstract

This independent study project is a preparatory work for the research--paraffin wax micro particle impacting dynamics and phase change behavior on quiescent paraffin wax liquid surface. The purpose for this independent study project is to produce paraffin wax particles with diameter 100 um both in their original color and dyed color. The PW micro particles were made by emulsion method.

Introduction

In addictive manufacturing, the coupled heat transfer and phase change phenomenon for metal particle impacting on molten pool is of great importance for improving the micro structure of deposition part. However, in-situ observation of the dynamics is not possible because of inaccessibility and opaqueness of the melt pool[1,2]. Therefore, a model system is used to visualize the phenomenon during the impact.

The first task is to find the appropriate material for the experiment.
The material should be transparent especially in liquid state. The density in solid state is larger than the density in liquid state, and the melting point is above room temperature(25 °C) and lower than 70 °C. This is because a perfect sphere is expected at room temperature so that it cannot be molten and the overheat of the pool is expected to achieve at sufficiently low temperature so that it would be safe to perform the experiment. Several polymers, Polycaprolactone, Ethylene-vinyl acetate, aliphatic polycarbonate-based thermoplastic polyurethanes are taken into account. However, they are either too expensive or not thoroughly studied. After further exploration, paraffin wax(PW) is found to be a better material. It is a common material, both widely used in academic and industry, and it is comparatively inexpensive.

Paraffin wax has a lot of application in both academic and industrial area. Paraffin wax is used as phase change material for heat management in electronic devices, candles and biological sample embedding.

The paraffin wax micro particles can be produced by emulsion. An emulsion is a dispersion of one immiscible liquid in a second liquid. Based on whether the oil phase or water phase is dispersed in other, they are classified as Oil in Water(O/W) or Water in Oil(W/O) emulsions[3]. Paraffin wax is a oil-based material Therefore to form
paraffin wax particles as dispersed phase in water, O/W emulsion is used.

The size range of PW micro sphere is about several nanometers to several hundred micrometers. For PW particles with diameter range between several nm to hundred nm, phase inversion emulsion can be used. For producing hundred um diameters dispersion, normal emulsion process can be used.

The heir size Paraffin wax sphere can be varied based on the surface tension between two phases. The specific size can be achieved by tuning several parameters[4]. The parameters that alter the dimension of PW sphere are temperature, operating time, the amount of surfactant and the stirring rate. In general, the diameter of paraffin wax are related to the surface tension at the interface of the water phase and the oil phase. As described in formula 1[5]:

$$\Delta p = \sigma \frac{2}{R}$$  \hspace{1cm} (1)

where $\Delta P$ is pressure difference between two phases, $\sigma$ is surface tension and $R$ is diameter of micro particle.

Therefore, to create paraffin wax with specific diameter, the surface tension need to be tuned. Surface tension can be changed by the temperature and the concentration of the surfactant. The surface tension decrease when increase the temperature, which is derived in the experiment. The surface tension decrease with increase the
concentration of the surfactant as shown in formula 2[6].

\[
\left( \frac{\partial \sigma}{\partial \Gamma} \right)_{T<0} < 0
\]

(2)

Where \( \sigma \) is surface tension, \( \Gamma \) is concentration of surfactant.

Surfactant can decrease the surface energy at the interface. In this experiment, because the water is the continuous phase, so the aqueous surfactant is used.

Surfactant is chemical compound with hydrophobic tail and hydrophilic tail. When the concentration is not high enough, the surfactant would aggregate at the interface, with hydrophobic tail facing toward oil phase and hydrophilic tail toward water phase. When the concentration of surfactant in solution is higher than critical micelle concentration , at which surfactant saturate at interface, the hydrophobic tails of surfactant would combine together and hydrophilc tails head toward water as shown in Figure 1. This aggregation is called mecelle. This is the mechanism how the emulsion forms.

![Figure 1](image_url)
Experimental procedure

Material

Paraffin wax with melting point 53-58 °C was purchased from Sigma-Aldrich, Inc., Sodium dodecyl sulfate(SDS) was purchased from Sigma-Aldrich, Inc.. The candle dye was purchased from the Candlewic Company.

Experimental procedure

The parameter chosen in this experiment is based on previous studies[8]. To produce the paraffin wax, a hot plate with magnetic stirrer was used as shown in Figure 2. First, a beaker with 250ml water was placed on the hotplate. Then 50ml Sodium dodecyl sulfate(SDS) solution with 6 wt% concentration was then slowly poured into water. The temperature of solution was controlled at 80 °C. After sufficient mixing, 4.2 g molten paraffin wax was poured into the solution. The stirring process with stirring rate of 400 rpm last for 2 hours. Then the paraffin wax was cooled at room temperature. The paraffin wax produced after cooling process is shown in figure 2.
There are paraffin wax particles with various diameter at the surface of the bulk paraffin wax sample. They were taken off the chunk by tweezer.

The paraffin wax particle created were varied in size. The smallest particle has the size 100 um and biggest one has diameter 2 mm. The diameter of the particle was measured under optical microscope.
Further the dye was used for creating paraffin wax particles with color. The dye was added in molten paraffin wax and then mixed thoroughly in the oven.

![Image of dye in molten paraffin wax](image)

**Figure 4. navy blue dyed molten paraffin wax**

The further work for this project is to conduct the particle impacting experiment with the dyed paraffin wax sphere and characterize the impacting and melting dynamics using high speed camera.

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