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# High energy dry coating mixing: Elements on velocities, temperatures and melting

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## ABSTRACT

In dry coating processes no liquid, solvent, binder or water, is required. This makes them advantageous compared to wet coating alternative: being low cost, no drying step required, environmentally friendly (no volatile organic compounds related problems) and simple in operation. But there is still a lack of fundamental knowledge of the implied mechanisms. Often the process itself has been empirically designed and is not always well characterized.

In this work, some hydrodynamic elements are developed in a high energy mixer used for dry coating: the specific behavior of the Nara hybridizer NHS-0 is investigated. In the first part, recirculation velocities and temperatures are derived during the process, or when the purge is on. The effects of the pressure purge and of the rotor velocity on the purge and the effect of the rotation on the temperature increase are studied. In the second part, a simple experimental approach is carried out to derive qualitatively the local temperature inside the apparatus with the help of polymers with different melting temperatures. This approach illustrates a first step for dry coating application with waxes.

### Keywords:

Dry coating  
Hybridizer  
Recirculation velocity  
Temperature increase  
Melting

## 1. Introduction

In dry coating processes, the core (host) particles are coated with fine (guest) particles by mechanical forces, creating composite powders with improved or new properties. During the operation occur de-agglomeration, ordered mixing, impact and shearing and eventually specific interactions according to the compatibility of the studied powders (electrostatic forces, van der Waals, embedding, mechanical interlocking. . .) [1]. Fig. 1 illustrates the various composite structures that can be obtained according to the equipment, the powder characteristics, the process conditions including processing time and weight fraction of guest to host particles.

In this work, the Hybridizer NHS-0 from NARA Machinery is studied from an hydrodynamic point of view. The recirculation and purge process are investigated as well as the influence of the rotation on the local temperature inside the mixing chamber.

## 2. Materials and methods

### 2.1. Process

The Hybridizer from Nara Machinery is a high energy mixer that has been often used in the literature to carry out dry coating [2–4].

It is composed of a stator (flat vertical cylindrical vessel jacketed for either cooling or heating) a rotor (close to a 6 blade Rushton turbine) and a re-circulating tube (Fig. 2). Due to high centrifugal forces, particles move to the upper part of the rotating chamber and recycle continuously till the end of the process. At the end of the operation, the powder is taken out of the system with a pressure controlled purge. It corresponds to a valve and a manometer that can be manually adjusted between 0 and 0.3 bars. When the valve is opened, an exit valve is simultaneously opened (see Fig. 2). The air suddenly blows inside the chamber and pushes the powder out of the system through the exit to a bowl and a fabric filter. The energies inside the mixing chamber can be estimated rapidly from the paddle end velocity [5], but the velocity information is not directly known in the re-circulating tube.

The system also has a temperature sensor, which monitors the temperature developed inside the mixing chamber during the operation. Without controlling the cooling jacket, there may be a rapid and intense temperature increase. This should be avoided for thermo-sensitive powders (degradation, phase transition or even melting).

### 2.2. Measurement device

The measurement system is a thermo-anemometer 'testo 425' that can measure the temperature in the range  $-20$ – $70$  °C and the velocity of air in the range  $0$ – $20$  m/s. Preliminary analysis showed no difference in the average data with the two tested integration times: 10 s and 2 min.

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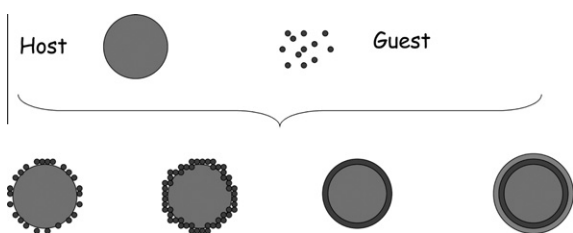


Fig. 1. Monolayer discrete, continuous (porous), continuous (film), and multilayer dry coating results.

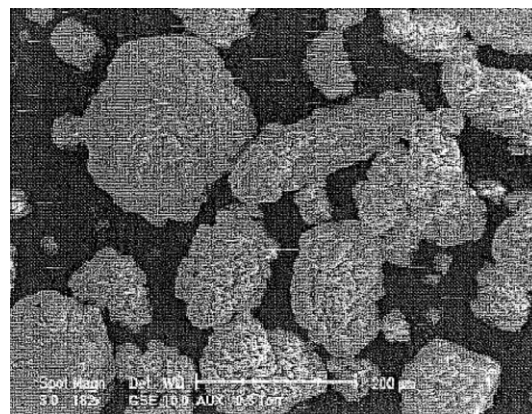


Fig. 3. SEM picture of used PEG 100000 polymer powder.

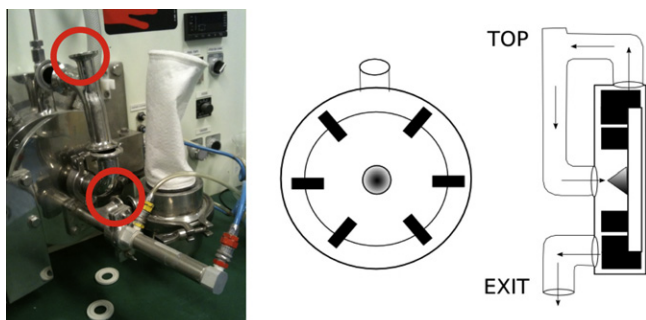


Fig. 2. NARA Hybridizer, with indication of 'top' and 'exit' positions.

### 2.3. Experimental procedure

The velocities and the temperatures are measured in the upper part of the system ('top') and in the bottom part of the system ('exit') seen in Fig. 2. For these measurements, there are no particles in the systems. This will be discussed further in the paper.

To study the influence of the rotation on the temperature evolution, two ways are used. In the first method we follow the temperature of air inside the re-circulating tube, with or without temperature control from the cooling jacket. In the second one, we qualitatively study the evolution of different polymers inside the chamber, i.e. if they melt. The powders are different PEG polymers (from Fluka): PEG 1500, PEG 35000 and PEG 100000 with respective melting temperatures of 50, 65 and 85 °C (Fig. 3).

The atmospheric conditions were checked for every experiments. The changes of temperature and relative humidity are tempered in the laboratory and are corresponding to maximum variation of air moisture of 3–4%.

## 3. Results and discussion

### 3.1. Velocity study

When the system is working without air purge, the rotation of the blades creates an air flow through the re-circulating tube and a flow through the exit tube that are measured and shown in Fig. 4. The larger the rotation, the larger the re-circulation, as expected. The configuration of the exit tube (perpendicular to the rotation plane) explains probably the smaller velocities for  $V_{exit}$  compared to  $V_{top}$  (tube tangential to the rotation plane). From these information, it is possible to derive the maximum entrainment particle diameter in the system.

These results can be compared with the simulation results from Mujumdar [6], and show good agreement. For example, the Fluent simulation in single phase give at 5000 rpm velocities in the re-circulating tube in the range of 5–15 m/s. Moreover it is fairly uniform in that tube, except when the fluid leaves the mixing chamber and enters the tube. If particles are in the system, the

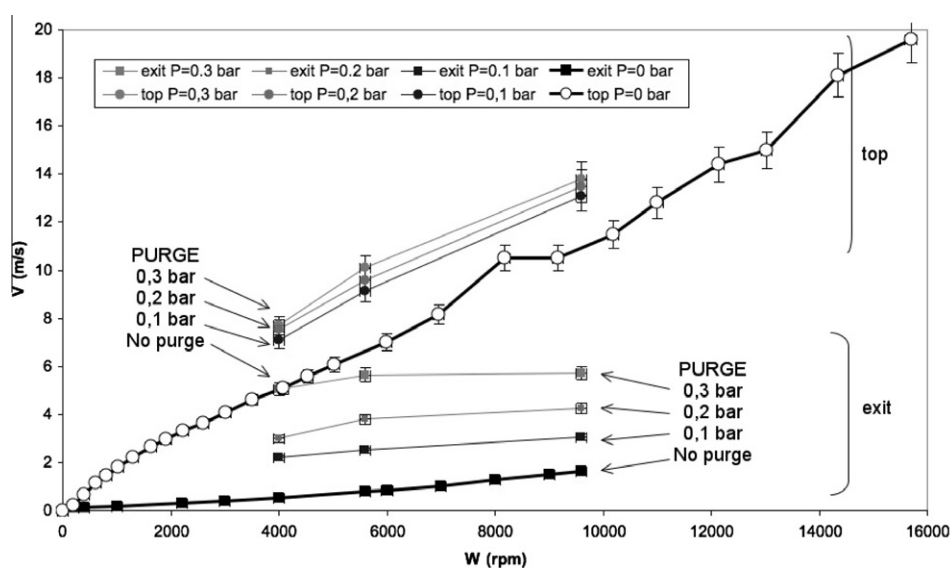


Fig. 4. Velocity variation with rotation in 'top' and 'exit' positions (no jacket cooling).

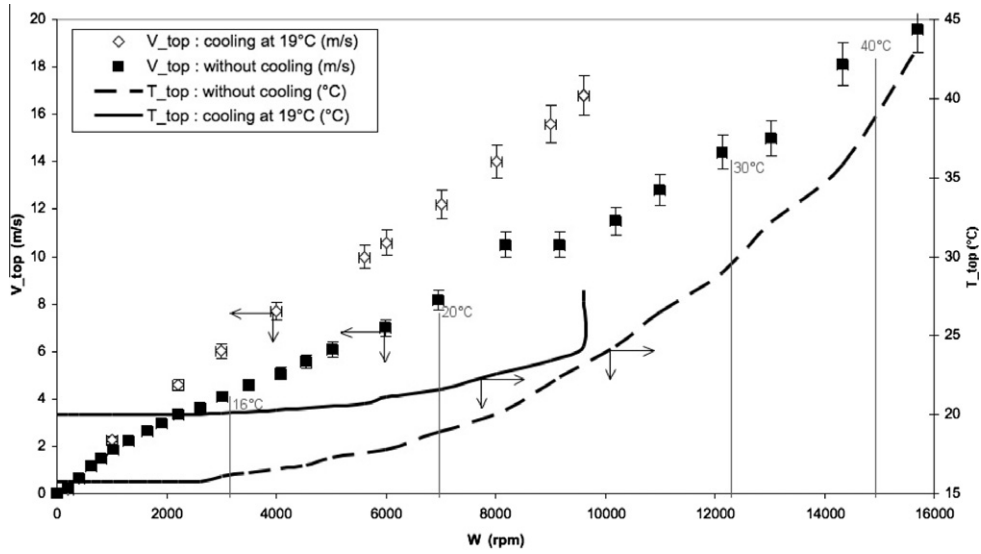


Fig. 5a. Evolution of the 'top' temperature and velocity with or without cooling jacket.

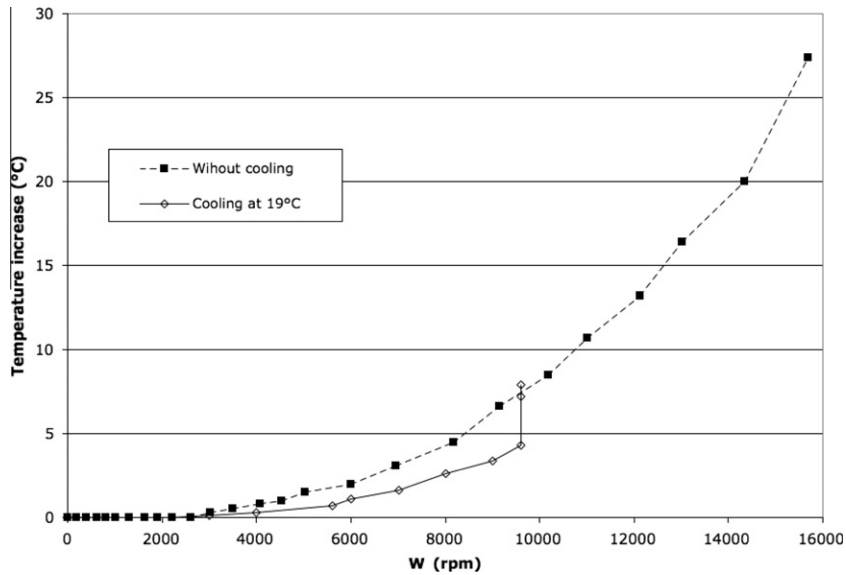


Fig. 5b. Evolution of the 'top' temperature increase with or without cooling jacket.

multi-phase simulation shows that the velocity decreases by approximately 10% in the volume-fraction range 0.03-0.1.

With the air purge, for the recirculation, the pressure has a moderate influence on the velocities compared to the rotation effect. For the exit tube, the rotation effect is insignificant compared to the pressure effect. As expected, this is the pressure which controls the purge of the powder out of the system.

### 3.2. Temperature study

The temperature measurements from the thermo-anemometer and from the thermal sensor of the Hybridizer were compared and are within the expected variability range, i.e. a few degrees. In a first set of experiments, the manipulation time was 6 min, which corresponds to the classical dry coating manipulation time. The jacket temperature control was set at 19 °C.

The effect of the rotation velocity is highlighted in Fig. 5a showing its combined influence on the recirculation velocity  $V_{top}$  and temperature in top position  $T_{top}$ . If we consider the latter influence, when the cooling jacket is not active, the temperature gradually increases with the rotation velocity and even reaches more than 40 °C at 16,000 rpm. The effect of cooling can be seen in Fig. 5b showing the variations of the temperature increase ( $T - T_{initial}$ ) with the rotation velocity. Thermal behavior comparison can be achieved on this graph, as it takes into account that the initial temperature conditions are different for the experiment with cooling (20 °C) and for the experiment without cooling (16 °C). Between 0 and 3000 rpm, during the experiment time, no temperature increase is observed. From 3000 rpm, a gradual heating occurs with increasing rotation when the cooling is off (expected) but also when the cooling is on (unexpected). A possible explanation is that the thermoregulation is not efficient. Up to 9600 rpm, temperature control is partial. But after 9600 rpm, the regulation system can't

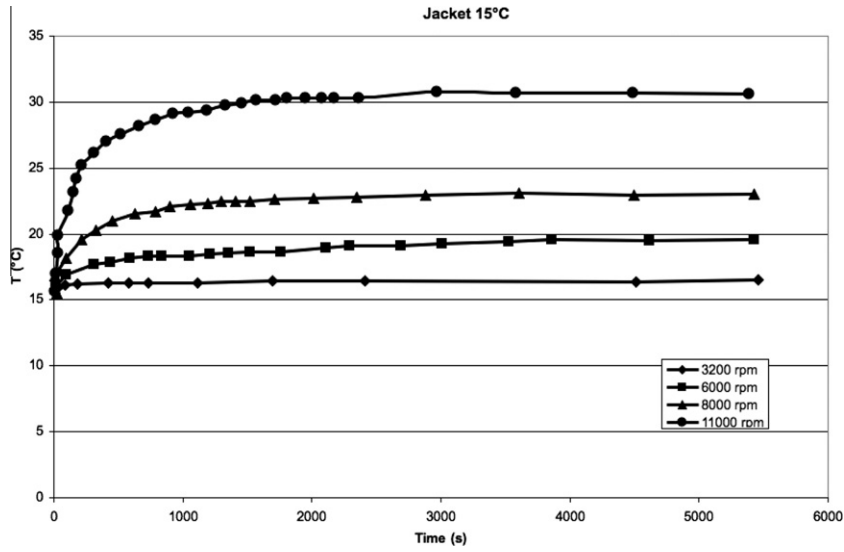


Fig. 6. Temperature–time profiles for cooling jacket temperature at 15 °C.

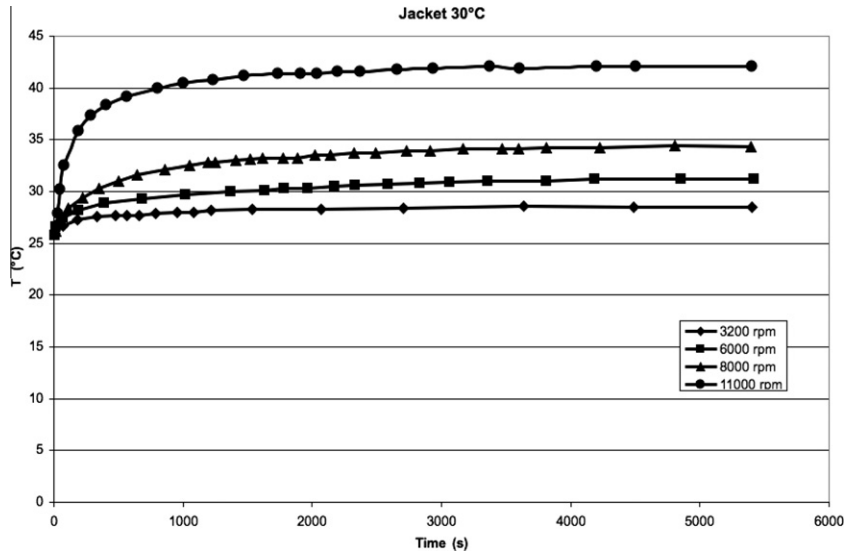


Fig. 7. Temperature–time profiles for cooling jacket temperature at 30 °C.

stand anymore heating. As a result, there is a steep variation of the temperature.

For the velocities, the observed difference could be explained by the difference in viscosity of the air circulating in the system: the induced flow is larger at higher temperatures corresponding to smaller viscosities.

Further experiments were carried out with 4 velocities (3200, 6000, 8000 and 11,000 rpm) and 4 jacket temperatures (15, 20, 25 and 30 °C). For 8000 and 11,000 rpm there is an extra jacket temperature of 13 °C. Figs. 6 and 7 show two time-profiles of the measured temperature up to 6000s for 15 and 30 °C jacket temperatures. It varies rapidly at the beginning, and for all the studied situations, after about 15 min, 90–95% of the limit temperature  $T_{\infty}$  is reached (equilibrium).

Fig. 8 represents the evolutions of the plateau temperature  $T_{\infty}$  with  $T_j$  for the 4 studied velocities. In all the situations, the evolutions are linear and the slopes are independent on the velocities. The data can be represented by the relation (1).

$$T_{\infty} = 0.8 T_j + b \quad (1)$$

In relation (1) the origin intercept  $b$  is dependent on the rotation velocity  $W$ .

Fig. 9 shows the relation between  $b$  and  $W$ . A linear fit satisfactorily represents the experimental data with a correlation factor of about 0.98.

In conclusion, in the investigated domain, simple linear regression leads to the Eq. (2).

$$T_{\infty} = 0.8 T_j + 0.0015 W \quad (2)$$

With:  $T_{\infty}$  is the equilibrium temperature,  $T_j$  is the jacket temperature, and  $W$  is the rotation velocity (rpm).

The relative evolutions are as follows: for a given jacket temperature  $T_j$  the larger the rotation  $W$ , the larger the  $T_{\infty}$  or for a given rotation  $W$ , the smaller the  $T_j$ , the smaller the  $T_{\infty}$ .

### 3.3. Melting study

In this part, 3 different PEG polymers were put in the hybridizer in rotation: PEG 1500, PEG 35,000 and PEG 100,000 (with respective melting temperatures of 50, 65 and 85 °C).

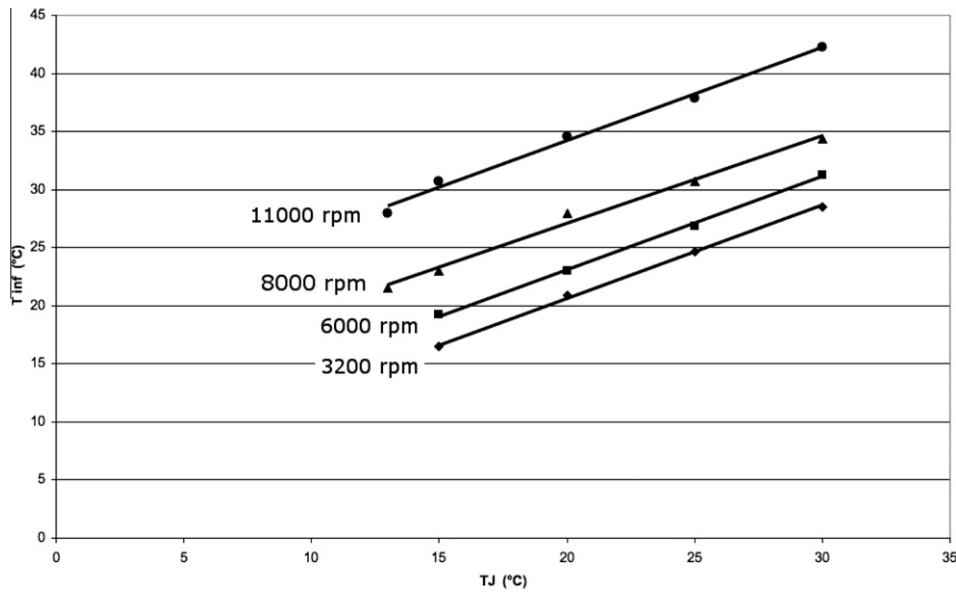


Fig. 8. Plateau temperature ( $T_{\infty}$ ) varying with  $T_J$  and  $W$ .

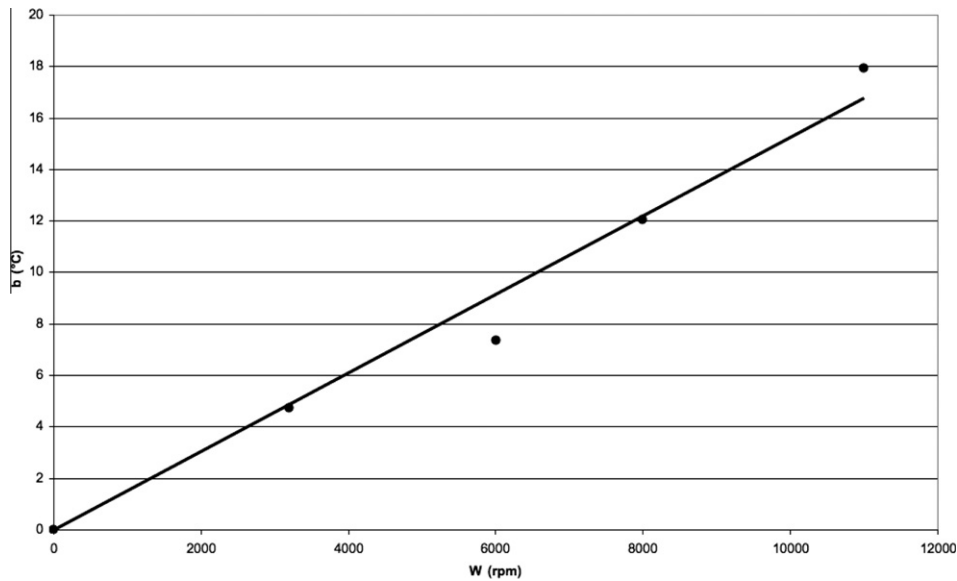


Fig. 9. Evolution of the origin intercept ( $b$ ) with the rotation velocity ( $W$ ).

The experiments used 30 g batch in the system at 1600 rpm, without cooling jacket. Due to friction mechanism (particle-particle and particle-walls), it was expected larger temperature increase of the system compared to previous one phase results. The procedure is to determine the time after which a polymer is melted. Knowing the fusion temperature of these polymers, it gives an estimation of the minimum temperature reached after that time.

The conclusions are that the larger the fusion temperature, the larger the time to reach melting, as expected. They are 6 s, 2 and 3 min, respectively for PEG 1500, 3500 and 100,000. Fig. 10 represents the evolution of the detected fusion temperature with the corresponding fusion time. This graph could be considered as qualitatively showing the evolution of the local temperature of the given system, considering similar situation with the three different polymers. There is a steep increase of temperature in the first seconds, then calm down after a few hundreds of seconds.

The experimental data could be compared with a simple model describing the temperature evolution for a given powder in the hybridizer in rotation. It is a first step for dry coating application with waxes, in order to know the minimum velocity or the minimum time to achieve dry coating with melting.

#### 4. Conclusion

This paper illustrates a preliminary study to characterize qualitatively the velocity and the temperature ranges inside the Nara Hybridizer, in the idea of dry coating applications with melting of the guest particles on the host particles (e.g. waxes). To have a more precise information, further manipulations are necessary (e.g. for the velocity, multi-phase tests). From these information, the generated data can be used for de-agglomeration, adhesion or heat exchange model or for comparison with simulation.

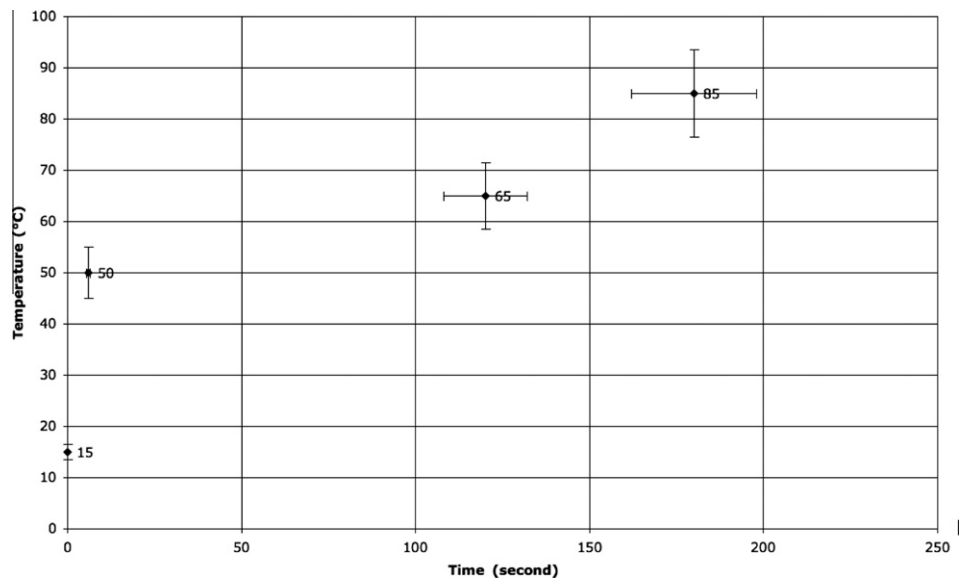


Fig. 10. Local temperature evaluation from polymer melting study.

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