

# EFFECT OF COLLOIDAL PARTICLES ASSOCIATED WITH THE LIQUID BRIDGE IN STICKING DURING DRYING IN SUPERHEATED STEAM

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**Abstract** It is important in the design of a drying system to evaluate the sticking behaviour of the materials being dried. A new approach to the sticking issue is applied in this study by carrying out a sticking test for the liquid associated with the materials under study. It was found that the liquid bridge is responsible for the initial sticking of the materials to the contact surfaces. The colloidal material in this liquid is eventually responsible of building a sticky solid bridge during drying. The glass transition temperature for the Brewers Spent Grain (BSG) particles and the colloidal solution expelled from these particles were tested using Differential Scanning Calorimetric (DSC). However, the chopped BSG particles showed no glass transition temperature; there were an appreciable number of particles stuck to the rotary drum dryer and the sample holders during drying. The colloidal particles in the liquid bridge were filtered and concentrated through evaporation and then analysed by DSC where they showed a glass transition temperature at (-23) and (-33) °C. In addition, the associated liquid thus prepared showed a honey consistency and a sticky touch when concentrated. These two properties are indications that this colloidal material may be responsible for sticking the BSG to the steel surfaces during drying.

**Keywords:** colloidal particles, sticking, liquid bridge, rotary drum, superheated steam drying.

چکیده در طراحی یک سیستم خشک کن، ارزیابی رفتار چسبندگی موادی که خشک می شوند پارامتر مهمی است. در این مطالعه با انجام تست چسبندگی برای مایع مربوط با مواد مورد بررسی، راهکار نوینی در ارتباط با این موضوع به کار گرفته شده است. بررسی ها نشان داده است که پل مایع، عامل چسبندگی اولیه مواد به سطح تماس بوده و همچنین ماده کلوئیدی موجود در این مایع عامل ایجاد یک پل جامد چسبنده در طی فرآیند خشک کردن می باشد. دمای گذار شیشه ای برای ذرات دانه ای مورد استفاده در آبیجو سازی (BSG) و محلول کلوئیدی حاصل از آن با استفاده از آزمایش های اختلاف کالریمتری (DSC) مورد بررسی قرار گرفته است. اگر چه ذرات خرد شده (BSG) دمای گذار شیشه ای را نشان نداده اند اما تعداد قابل ملاحظه ای از آن ها به خشک کن دوار و نگه دارنده های نمونه در حین خشک کردن چسبیدند. ذرات کلوئیدی در پل مایع فیلتر و با استفاده از روش تبخیر تغلیظ شد. همچنین با بهره گیری از آنالیز (DSD) دمای گذار شیشه ای در دماهای °C -23 و -33 نشان داده شده است. علاوه بر آن مایع آماده شده با روش بالا قوام و چسبندگی در حین تغلیظ از خود نشان داد. این دو پارامتر موید این مطلب بوده که مواد کلوئیدی عامل اصلی چسبندگی ذرات BSG به سطوح استیل در حین خشک کردن می باشند.

## 1. INTRODUCTION

Drum drying is an example where stickiness is an important issue. The drum equipment and parts are normally made of cast iron which has high surface energy ( $>71\text{mN/m}$ ). Surface energy is a measure of the solid surface wettability. Wettability is responsible for building a liquid bridge between two surfaces. The stickiness of food materials during drying is governed by a combination of both adhesive and cohesive phenomena. The interfacial surface energy of the equipment and the material plays a critical role in controlling adhesion, while the interface diffusion and viscoelastic behaviour control the cohesive phenomena [1].

Some foods exhibit a marked tendency to adhere to a contact surface, which is generally known as stickiness. Although the stickiness may have favourable processing attributes such as agglomeration, it is often a problem in manufacturing operations. Stickiness can cause lower product yield, operational problems, equipment wear, and fire hazards. Although the problem of stickiness in food industries has been recognized long before, methods developed to quantify it have been empirical in nature. There is no general compromise as to what factors and forces are involved in stickiness. Some authors attribute stickiness to adhesive forces, some attribute it to combined adhesive and cohesive forces [2], and others include viscosity [3] and liquid bridge [4].

During drying process the surface composition of the material can be different from the bulk material due to migration of solute to the surface of the particles. Therefore, the cohesive or adhesive behaviour of the particle surface cannot necessarily be the same as the bulk material being dried [5]. Adsorption of moisture by confectionary products results in lowering its viscosity that may lead not only to sticking, but also to the collapse of the product structure [2].

Mu and Su [4] illustrated that the liquid bridge between wet surfaces is several orders of magnitude larger than other adhesion mechanisms such as electrostatic and Van der Waals forces. In their study the rupture energy of a pendular liquid bridge between two spherical particles were

obtained. They found that the rupture energy increases with increasing liquid bridge volume.

The characteristic of interaction between a particle and a metal surface is strongly modified by the water meniscus. This meniscus results in an additional capillary force increasing the adhesion of the particles to the surface [6, 7].

The liquid bridge and the water meniscus, due to initial condensation, may be dominant at the beginning of drying, while the stickiness of the material after drying of the liquid bridge is mostly due to that described by Mori. Mori [8] found that the edge of the droplet receded during evaporation, and an incomplete ring-like and tiny spots of small particle aggregates were observed. These spots may integrate to be the core for the solid bridge. This phenomenon is the reason for our work in this paper to study the liquid associated with the particles.

Many researchers reported that the glass transition temperature and the sticky point temperature are the main factors in sticking. Ozmen and Langrish [9] found that the glass transition temperature was virtually the same as the sticky-point temperature measured using thermo-mechanical tests. Principal components present in foods are low molecular weight sugar and some organic acids. The food components have low glass transition temperature,  $T_g$ , and are very hygroscopic in their amorphous state. In addition, water acts as plasticizer decreasing the glass transition temperature [10].

Adhikari et al [11-14] developed a model to predict the moisture and temperature histories of fructose and lactose droplets. Adhikari has also predicted the development of surface stickiness of these sugar materials reasonably well. Lactose surface reaches a non-sticky state through crystallization or transformation to glassy state. Fructose droplets remained sticky even in the glassy dry state due to their low  $T_g$ . There is a clear indication of the dependence of the stickiness of powder materials with high sugar content on the glass transition temperature of amorphous sugar [15-17].

Many published articles deal with cohesion of powders, as quantified in terms of the so called the sticky point temperature. Many studies in this area [18-25] are more concerned with measuring cohesion between particles than adhesion. The

resistance to shearing motion in sticky point test is indicative of cohesive force among the powder particles and not the adhesive force between the particles and the equipment surface [12, 14, 26, 27]. It has been found that the glass transition temperature is more correlated to the bulk property of the materials than the surface property [28]. This was also another reason to study the liquid expelled from the particles.

In this study the property of the liquid film that covers the Brewers Spent particles due to the initial condensation are considered as the major factor in initiating sticking. The initial condensation happens on the surface of the particles and instantaneously the particle surface temperature increases to 100 °C. Then the moisture content of the first layer at the particle surface increases to equilibrate with the condensed water. These parameters affect the composition of the liquid at the surface. This liquid may form a liquid bridge which can react to give hydration products; these colloidal products (hydration products) can glue the surfaces together [29].

The small particles of nano-particles appearance in the liquid bridge may be formed due to friction and collision between the particles during the processing, such as in rotary drum dryers. Also these particles may be due to diffusion from the particle surface to the water film around the particle. In addition, migration of moisture from the interior of the particle may carry these colloids to the surface. The effect of particle size on sticking has been well studied in the literature. As the particle size reduces, the cohesion and the adhesion forces increase [30]. During drying, droplets of amorphous solution become concentrated at their evaporation surface and can change state from a viscous fluid to a rubbery and to a glassy state as the moisture content decreases [28]. This solid or glass bridge may be weak because its strength depends only on the total area of the junction between the particle and the surface.

Since the colloidal particles in the condensed water are different in size, this may create a different glass transition temperature. This will lead to a sequence of glass transition processes beginning with the lowest particle size in the colloidal mixture and finally lead to solid bridge due to their glass transition and melting point

depression. The liquid associated with the particles undergo three steps: liquid bridge, elastic bridge, and finally solid bridge. This paper is concerned with the sticking of the BSG particles to the rotary drum dryer. It was shown that the shell of the particles and the particles as whole do not go through glass transition temperature. This led to hypothesis that the liquid associated with the particles due to their initial moisture content or due to the initial condensation during drying is responsible for the particle sticking. To be more specific, the colloidal particles which have either dissolved or dispersed as fine particles in the liquid have a very low glass transition temperature, and as shown in this paper, this is the main factor in sticking.

## 2. PREPARATION OF SAMPLES AND COLLOIDAL MATERIALS

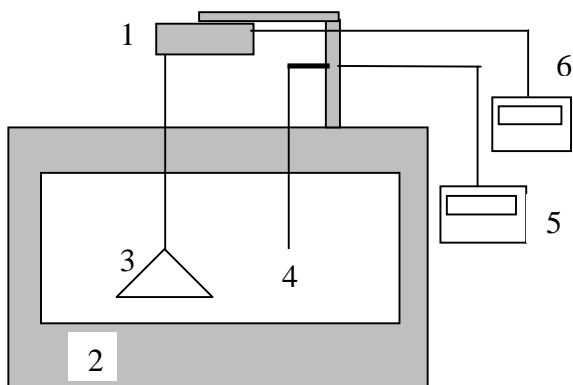
A domestic steam oven was used as a closed environment for preparation of samples. The oven was cubic in shape with inside dimensions of: 24 cm height, 40 cm wide and 45 cm deep. A thermal glass was fixed at the front of the oven to give a clear view. The schematic diagram of the steam oven is shown in Figure (1). During the experiments there was no condensation on the front glass from either side.

The oven was a Built-in steam oven model ED 220-100 by GAGGENAU. The temperature range was 30 to 230 °C and the steam percentages were set from 0, 30, 60, 80 and 100 %.

Two small holes were made at the top surface of the oven in the centre. One hole was made to insert the K-type thermocouple. This hole was sealed with high temperature resistance silicone to prevent air sucking or steam loses. The thermocouple was directly connected to a temperature display. A long steel wire was inserted into the oven from the second hole to hold the samples. This second hole was made a little bigger than the diameter of the wire to decrease the escape of steam from the oven. On one end inside the oven the sample was placed on a mesh tray and hooked to the wire, and on the other end the wire was connected to a load cell. The steel wire was 65 cm long, 5 cm inside the oven and 60 cm outside to make sure that the load cell was far away from the

hot surface of the oven which was also insulated by glass wool.

The load cell was fixed far away above the oven. A small fan was used to circulate air around the load cell to prevent any heat build up. The load cell was capable of reading weight force in the range 0.0 to 500 gm with the accuracy of one decimal digit. A digital display was used to show the reading of the load cell.



**Figure 1.** Schematic diagram of the steam oven system; 1- load cell, 2- steam oven, 3- triangular shape samples holder, 4- thermocouple, 5- temperature display, 6- weight force display.

The material under study was brewers spent grain, BSG. Many experiments were carried out to prepare samples of BSG. The differential scanning calorimetry (DSC) was used to measure the glass transition temperature. Four samples of different moisture content of the BSG were prepared first. Chopped BSG with a knife was placed in a tray connected to a load cell. Each sample weighed 20 g with initial moisture content of 75 % w.b (wet basis). These samples were placed in the tray and then dried under 100 % steams and 150 °C to different moisture contents of 15, 25, 35, and 45 % (w.b).

Another two sets of samples were prepared, this time by extracting the colloidal particles from the chopped BSG. The samples were prepared by mixing 20 g of the BSG with 100 mL of distilled water at 70 °C. These sets of samples were prepared in order to mimic the drying under steam, while the material gets boiled and its temperature rises to 100 °C due to initial condensation. Then, the samples were mixed by a magnetic stirrer for 1

minute. The mixture was then sieved by a 25 and 8 micrometer mesh to ensure that the colloidal particles in the collected water solution were less than 25 and 8 micrometers respectively. The solution was then collected and concentrated by evaporation with continuous stirring and heating at 40 °C. The concentrated solution had a honey appearance and a sticky touch. Finally the samples were placed in an oven at 40 °C to prepare materials with different moisture contents. The colloid materials were identified according to their moisture content on wet basis, Kg solid/ Kg sample. The moisture content of the two sets of samples was 45% and 21 % w.b respectively.

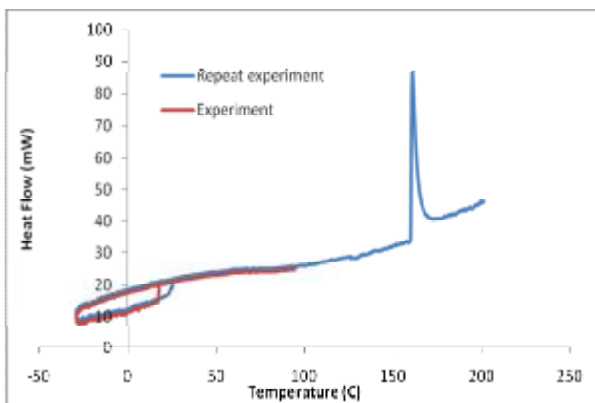
A Perkin-Elmer Differential Scanning Calorimeter (Pyris 1) was used to determine the glass transition temperature with a scanning speed of 10 °C/minute. Each experiment was repeated twice to better validate the results. The ranges of temperatures were -50 to 200 °C. All the experiments were carried out twice, the first test in the temperature range between [(-20°C)-(-30°C)] and [(90°C)-(105°C)] to ensure that the samples were not damaged. The second test was done in a wider range, between [(-30°C)-(-50°C)] to [(200°C)-(230°C)].

## 2. RESULTS AND DISCUSSIONS

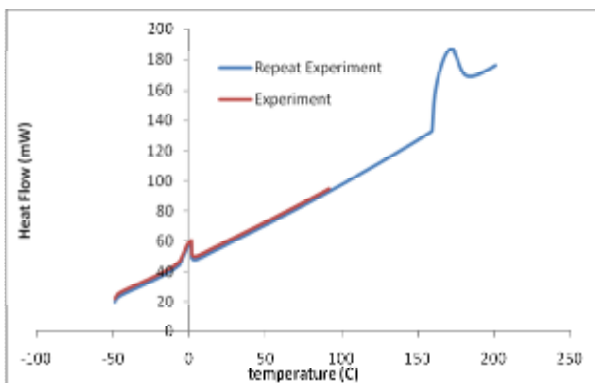
During drying of BSG in a rotary drum dryer under superheated steam environment some of the materials stuck to the surface. The most severe sticking happened at the front of the drum near the feeder. After stopping the feeding and turning off the drum, it was found that these materials when collected from the surface were slightly stuck together. Also, during drying the samples on the steel trays for the DSC test, the grains showed slight stickiness to the steel tray. The liquid bridge that formed due to the initial condensation was obviously responsible for the initial sticking of the particles.

The all chopped BSG at different moisture contents did not show any change in heat flow during the test in the DSC. The DSC scans are shown in Figures 2, 3, 4, and 5 for 15, 25, 35 and 45% moisture contents (w.b) respectively. The period where it is expected to have the glass transition temperature for the 45% M.C is plotted

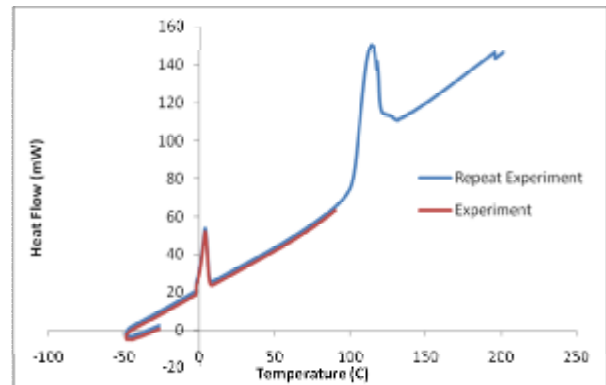
in Figure 6. There was absolutely no change in heat flow, suggesting that the material had no glass transition temperature. The only sample that showed a peak of crystallization was at 45% w.b. moisture content, however the peak was due to the water evaporation. These results were expected because the shell of the BSG is mostly fibers that undergo decomposition rather than glass transition or melting. All the experiments were repeated twice after cooling the samples to room temperature for more accuracy. After finishing the test in the DSC, the samples containers were disclosed and a complete decomposition was observed. Figure 7 shows the trend for one of the samples (45% M.C) after it decomposes. The figure clearly shows no change in the heat flow despite the wide range of the test temperature [-50°C to -250°C]. Because most of the spent grain is made of fibres (70%) [31, 32], this may confirm the decomposition of the samples.



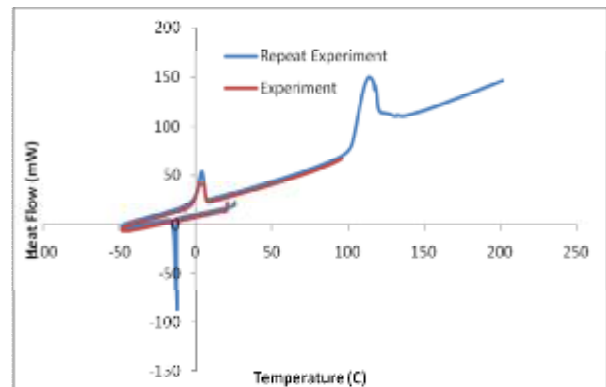
**Figure 2.** DSC results for the chopped BSG samples, the whole grain with a moisture content of 15 % w.b.



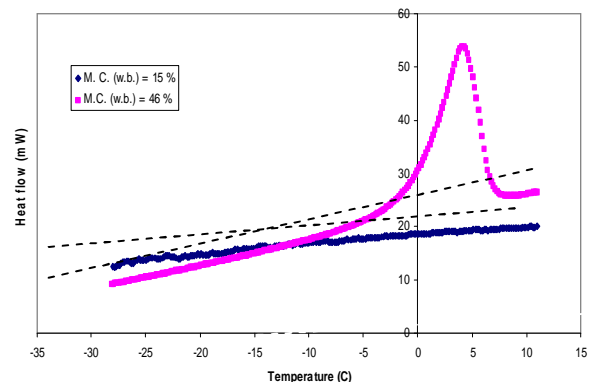
**Figure 3.** DSC results for the chopped BSG samples, the whole grain with a moisture content of 25 % w.b.



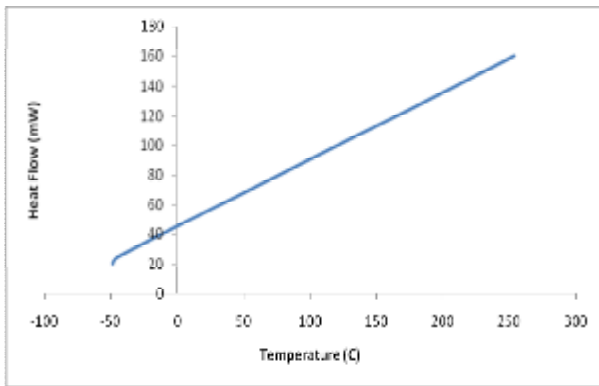
**Figure 4.** DSC results for the chopped BSG samples, the whole grain with a moisture content of 35 % w.b.



**Figure 5.** DSC results for the chopped BSG samples, the whole grain with a moisture content of 45 % w.b.

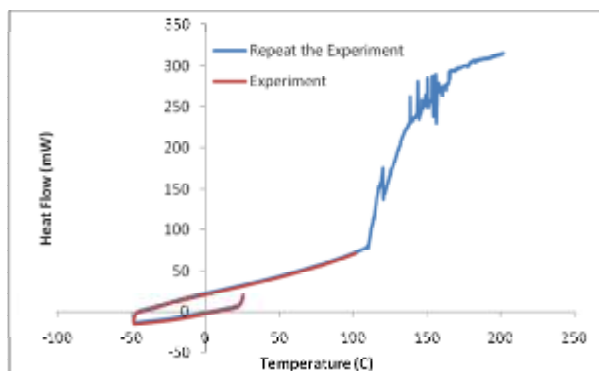


**Figure 6.** DSC results for the chopped BSG samples for the period where the glass transition temperature is expected, the whole grain, two M.C 15 and 45 % w.b.

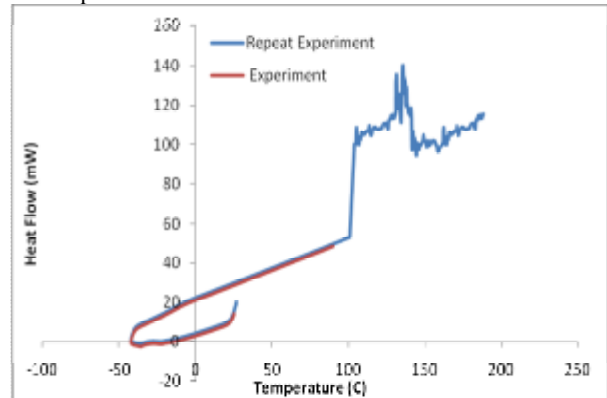


**Figure 7.** DSC results for the burned chopped BSG samples, the whole grain with 45% w.b.

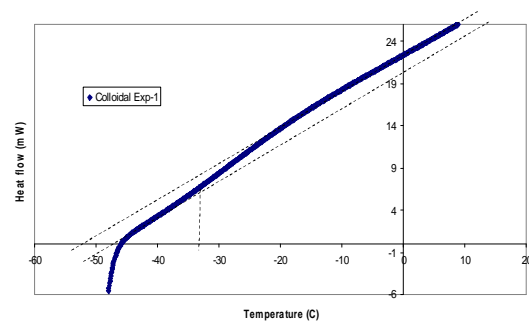
The 45% M.C colloidal samples with two particle size of 25 and 8 micrometer were tested and the results are shown in Figures 8, 9 and 10. Figure 10 is the enlarged part where the glass transition temperature appeared. The figures show that the glass transition temperatures of the samples were approximately around  $-33^{\circ}\text{C}$ . The slight slope of the DSC trend may be due to the range of the particle size present in the solution. Different particle sizes have different glass transition temperatures [30]. The range of the temperature where the glass transition  $T_g$  appears are between  $-33$  and  $-22^{\circ}\text{C}$ . The peaks above  $100^{\circ}\text{C}$  shown in Figures 7 and 8 were due to moisture evaporation from the samples, this then followed by higher peak where the decomposition happened. This material did not show crystallization or a clear melting point, perhaps due to the vast particle size range present in the mixture.



**Figure 8.** DSC results for the BSG samples, 25 micrometer colloid particles with a moisture content of 45 % w.b.

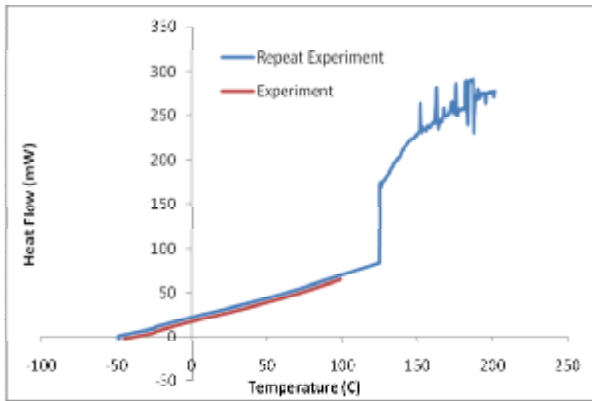


**Figure 9.** DSC results for the BSG samples, 8 micrometer colloidal particles with moisture content of 45 % w.b.

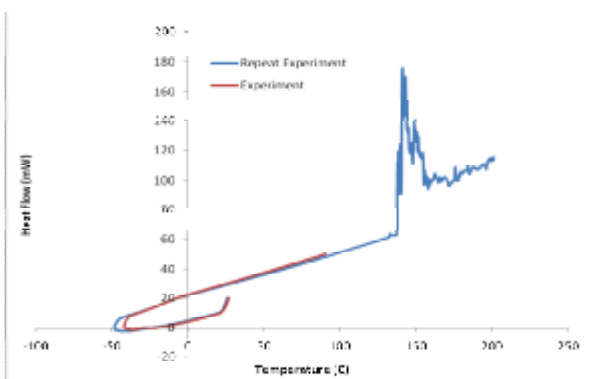


**Figure 10.** Magnified DSC results for the BSG samples, 25 micrometers colloidal particles with moisture content of 45 % w.b.

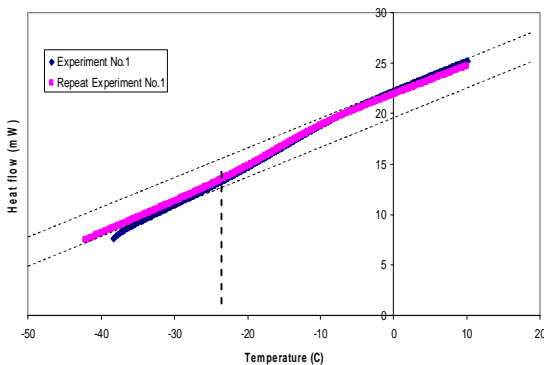
The second set of the colloidal samples were dried to 21% moisture content and two particle sizes of 25 and 8 micrometer. The test results are shown in Figures 11, 12 and 13. This sample showed a higher glass transition temperature due to its lower moisture content [10]. This experiment was also repeated twice after reaching 90 to  $100^{\circ}\text{C}$  and it showed the same trend as shown in Figure 13. The glass transition temperature appears in the temperature range  $-23$  to  $-9^{\circ}\text{C}$ . Because of the low moisture content of the colloidal samples, it did not show any peak due to evaporation; however it showed the melting peak followed by decomposition.



**Figure 11.** DSC results for the BSG samples, 25 micrometers colloidal particles with moisture content of 21 % w.b.



**Figure 12.** DSC results for the BSG samples, 8 micrometers colloidal particles with moisture content of 21 % w.b.



**Figure 13.** Magnified DSC results for the BSG sample, 8 micrometers colloidal particles with moisture content of 21 % w.b.

### 3. CONCLUSION

The glass transition temperature was selected as the indication of the sticking behaviour of the

materials. The chopped BSG samples showed no glass transition temperature as determined by the DSC analysis for different moisture contents of the materials. However, these materials demonstrate high sticking to the rotary drum surface and the basket used for drying the samples. The sticking force between the particles and the drum surface was low and the material could be removed by a small force. The concentrated liquid expelled from the BSG particles showed a clear glass transition temperature at different moisture contents and particle sizes. These results show that the colloidal particles may be responsible for this sticking behaviour.

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