

**DESIGN AND FUNCTIONING OF LOW PRESSURE  
SUPERHEATED STEAM PROCESSING UNIT**

by

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## ABSTRACT

Drying of distillers' spent grain (DSG) is needed to reduce both moisture content and mass of the material for extended storage time and reduced transportation cost. Superheated steam (SS) drying of DSG is a more energy efficient alternative to conventional hot air drying. Burning and lowering the quality of the food product can be a problem for temperature sensitive material, such as fruits and vegetables, due to the high temperature required in SS drying. A solution is to create a drying environment having a sub-atmospheric pressure, which is often referred to as low or vacuum drying. In this case boiling point of water is lowered and product can be dried at a relatively lower temperature.

Superheated steam drying is a fairly new technology. There is no study reported on drying DSG with SS under low-pressure and no system has been built for that purpose. To study the drying kinetics of DSG dried with SS under low pressure, it is necessary to custom design and construct a SS drying system that can dry product under low pressure. Therefore, the objective of this study was to design, fabricate, and test a SS drying system that could operate at sub-atmospheric pressure for drying DSG.

A custom designed superheated steam sub-atmospheric pressure (also referred to as low pressure) drying system was constructed and tested. Distillers' spent grain was successfully dried using the system under various levels of temperature from 95 to 115°C and pressure of either -25 or -20 kPa, with a SS velocity from 0.100 to 0.289 m/s.

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## 1 INTRODUCTION

Distillers' spent grain (DSG) is the nonfermentable residue left in distilleries from the production of ethanol fuels after fermentation. Fermented grains used in distillation include corn, wheat, barley, rice, and rye (Wampler and Gould 1984; Woods et al. 1994). During fermentation, most of the starch in the raw materials is removed and converted into alcohol and carbon dioxide. The reduction of starch increases the concentration of protein, fat, and fibre in DSG by three fold. (Kissell and Prentice 1979, cited by Tang et al. 2004; Wampler and Gould 1984). Therefore, DSG is an excellent source of protein and energy, which makes it a common source of animal feed for cattle and poultry. Also, DSG can be used as a flour supplement to produce food products for humans, including baked foods, extruded snacks, and baby foods (Rasco et al. 1990; Wu et al. 1984).

Spent grains usually come in the form of a liquid and solid mixture, having a moisture content ranging from 75 - 85 % wb (wet basis) (Tang and Cenkowski 2001). Mechanical de-watering using a screw press or centrifuge can only reduce the moisture content to 60 - 70% wb (Woods et al. 1994). Such high moisture in spent grain greatly contributes to mass volume, which is undesirable in transportation, and also causes the grain to spoil easily. Therefore, to reduce the mass and extend the storage life of DSG, it must be dried to about 10 % wb moisture content (Woods et al. 1994). Conventionally, DSG is dried using a drum or rotary drier using an inlet temperature of 500 °C with an outlet temperature of 115 - 120 °C. Hot air drying is, however a very energy intensive operation (Tang 2002). To reduce energy consumption, improve scent, odor and other

physical properties of the product, which are important to food industry, superheated steam can replace hot air in the drying operation of DSG.

Conventional hot air drying is being widely used in the food industry. However, the quality of product dried using hot air is greatly reduced from that of the original in terms of shrinkage and glass transition temperature (Ratti 2001). Furthermore, there are some potential risks associated with hot air drying, which can cause burning of products and explosion hazards. In addition, conventional hot air drying alone accounts for about a quarter of the total energy used in the food processing industry in Canada (Drouet 1984, cited by Woods et al. 1994). Therefore, researchers have started to look for a more efficient drying method as an alternative to hot air drying.

Although the concept of using SS as the drying medium in place of hot air was first proposed over a century ago (Elustondo et al. 2002), this technology has only emerged as an alternative and gained acceptance in very recent years. After the “oil crisis” in 1973, the publishing rate on steam drying substantially increased (Wimmerstedt 1995). Recently, SS drying has become more acceptable due to the recognition of its low energy consumption and short drying time, high drying rates, better quality products and reduced environmental impact in comparison to hot air drying. Superheated steam drying has been applied in various areas on the pilot, laboratory, and industrial scale (Zielinska et al. 2009). Food products dried using SS include green tea, sugar beet pulp, shrimp, potato chips, fries, cacao nuts, wheat, flour, vegetables, noodles, herbs, and spices (Pronyk et al. 2004; Caixeta et al. 2002; Prachayawarakorn et al. 2002; van Deventer and Heijmans 2001; Tang and Cenkowski 2000; Urbaniec and Malczewski 1997; Jensen 1992; Akao 1983, cited by Shibata and Mujumdar 1994). Other products such as lumber,

paper, coal, and sludge have also been dried using SS (Clayton et al. 2007, cited by Kudra and Mujumdar 2002; Potter and Beeby 1994; Woods et al. 1994; Cui et al. 1986).

Despite all of the mentioned advantages of using SS as a drying medium, the technology does not apply to materials that are temperature sensitive, such as fruits and vegetables. A solution to the problem is to create a drying environment that has a sub-atmospheric pressure, which is often referred to as low pressure drying or vacuum drying. When exposed to low pressures, the boiling point of water becomes lower, which allows the product to be dried above saturation point but at a relatively lower temperature. Superheated steam drying in vacuum causes less damage to the product, which helps produce better quality food products. In the food industry, the use of vacuum drying causes the removal of moisture from food to occur under low pressure which lowers the oxygen level. Browning due to oxidative reaction is reduced in the final product (Jaya and Das 2003). As a result, the combined use of superheated steam at low pressure can be advantageous.

The SS drying technology is still fairly new in the industry. The number of major dryer manufacturers that provide superheated steam drying equipment is limited (Kudra and Mujumdar 2002). Also, drying of DSG with superheated steam has only been studied by a few researchers. There are no studies of drying DSG with superheated steam under low pressure found in the literature. Therefore, to study the drying kinetics of DSG dried using superheated steam under low pressure, it is necessary to custom design, fabricate, and test a superheated steam drying system that can operate below atmospheric pressure.

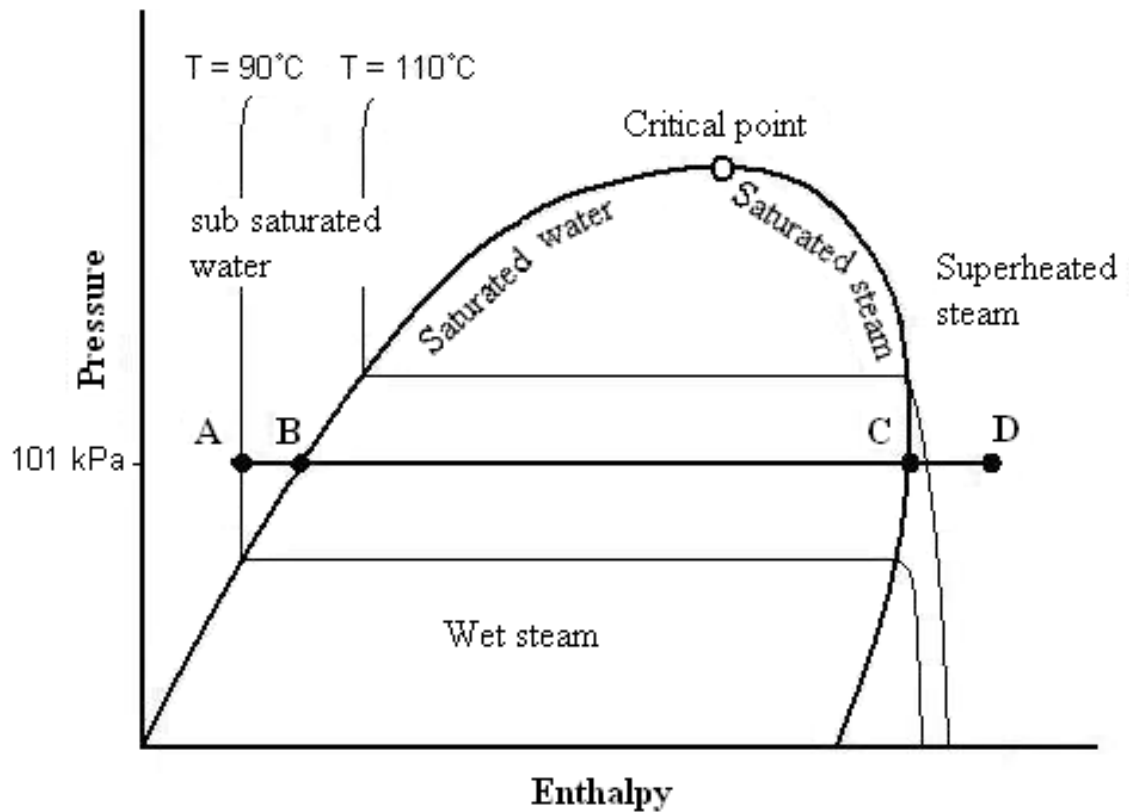
The objective of this study was to design, fabricate, and test a superheated steam drying system that could operate at sub-atmospheric pressure for drying distillers' spent grain.

## 2 LITERATURE REVIEW

### 2.1 Nature of superheated steam

Superheated steam is obtained when wet steam is heated at constant pressure and its temperature rises higher than the saturation temperature (refer to Figure. 1). Superheated steam can also be generated by lowering the pressure of wet steam below that achieved at saturation. The amount of superheat added to steam at constant pressure and the excess heat obtained at pressure lower than saturation pressure can be used in direct heating and/or drying (van Deventer and Heijmans 2001). In the case of drying, SS cannot undergo a phase change (condensation) and its temperature must stay higher than the saturation temperature throughout the drying time. In figure 1, point A represents water at 90 °C and 101 kPa pressure. As heat is added to water at a constant pressure, temperature increases to 100 °C and reaches point B. As additional heat is added, more liquid water changes to vapor state and reaches point C. At this stage, all water has evaporated and turned into vapors, producing saturated steam at 100 °C. Further addition of heat results in SS at the same pressure but higher temperature. Point D represents SS at 130 °C

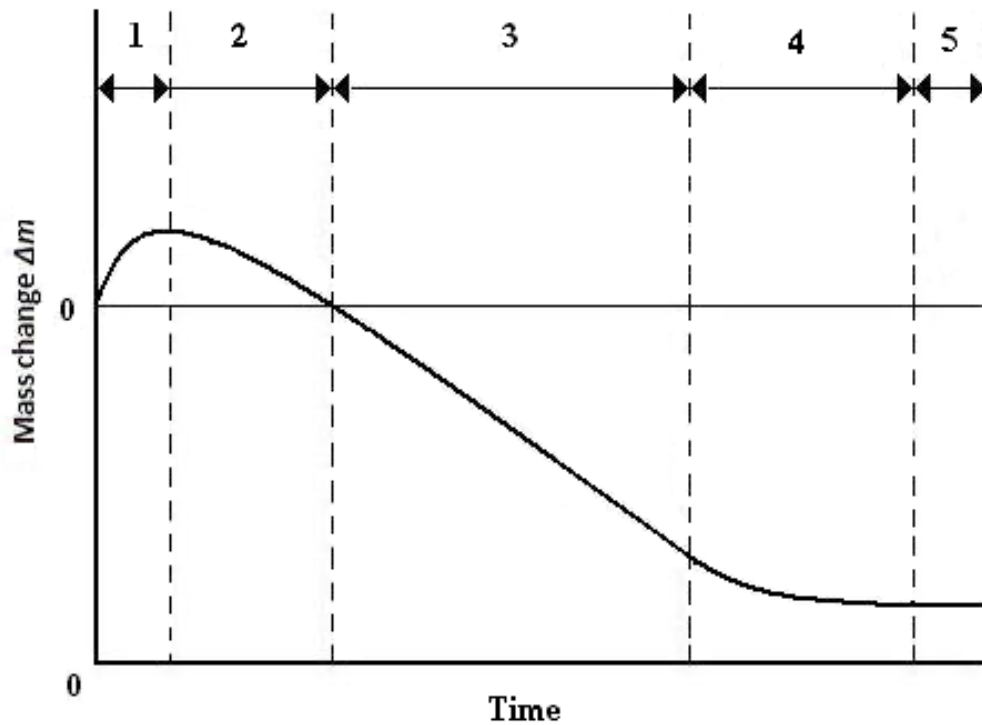




**Figure.1 Pressure-enthalpy diagram for steam-water and vapor. Line AD: Path of water at 90°C at atmospheric pressure to superheated steam (Adapted from Straub and Scheibner, 1984; cited by Singh and Heldman 2001).**

## 2.2 Superheated steam drying process

Superheated steam drying utilizes superheated steam as a drying medium to remove moisture from a material. Drying is achieved when SS transfers its sensible heat to the material and evaporates all or most of the moisture within the material. The SS drying process for a high-moisture-content material can be described in five periods: (1) Condensation period; (2) restoration period; (3) constant-rate period; (4) falling-rate period, and (5) hygroscopic period (Figure 2).



**Figure 2. The drying curve of superheated steam drying process. 1: Condensation period; 2: Restoration period; 3: Constant-rate period; 4: Falling-rate period; 5: Hygroscopic period (Adapted from Iyota et al. 2001b).**

### 2.2.1 Condensation period

Van Deventer and Heijmans (2001) suggested that it was possible to keep steam superheated after being introduced to the cold material being dried by balancing the drying rate with respect to the SS input amount. However, many authors (Zielinska et al. 2009; Cenkowski et al. 2007; Devahastin et al. 2004; Pronyk et al. 2004; Markowski et al. 2003; Kudra and Mujumdar 2002; van Deventer and Heijmans 2001; Iyota et al. 2001a; Tang et al. 2000) concluded based on their experiments that a certain amount of moisture gain was observed during the initial stage of processing with SS due to condensation. These results agreed with the drying curve of SS drying process (Figure 2).

At the start of the drying process when the material temperature was below the saturation temperature, enough heat was removed from SS contacting the material and caused the temperature of steam to drop below saturation temperature. This caused steam to condense onto the material surface. Therefore, the material gained moisture and a mass increase was observed.

### **2.2.2 Restoration period**

As the temperature of the material rises above the saturation point, condensation ceases. The material stops gaining mass (moisture), and evaporation begins, thus the start of a restoration period. When all the additional moisture is evaporated and the mass of the material falls to its original mass, the restoration period ends and a constant-rate period begins.

### **2.2.3 Constant-rate period**

Although evaporation has already started before this period, the drying of material only begins at this stage because additional moisture from the condensation period must first evaporate. When the initial moisture content of the material is greater than its critical moisture content, the moisture removal from the surface of the material should be at the same rate as that from the free surface of water, provided that the drying conditions are constant (Pabis et al. 1998). This is because the material surface is maintained at a saturated condition due to the moisture movement from the interior of the material to its surface. Therefore, the rate of drying is constant.

#### **2.2.4 Falling-rate period**

When the initial moisture content is lower than the critical moisture content, the falling-rate period begins, because the rate of drying decreases with the time of drying. At this stage, the saturated surface area of the material starts decreasing. The internal water transfer resistance of the material increases and rises to a point where it exceeds the external water transfer resistance from the material, thus lowers the rate of drying (Pabis et al. 1998).

#### **2.2.5 Hygroscopic period**

The hygroscopic period begins when the equilibrium between vapor pressure in the SS and vapor pressure in the solid (dried material) is reached, which the drying rate is brought to zero. In this stage, the material temperature rises to that of the SS (Elustondo et al. 2001). In addition, the material moisture content reaches the equilibrium moisture content for the given drying conditions and the drying process stops (Tang 2002).

### **2.3 Advantages of using superheated steam**

There are several advantages of using SS as a drying medium. The idea of using SS as an alternative to hot air in drying was first proposed in the book “Drying by Means of Air and Steam” by a German scientist, Haubrand, over a century ago (Elustondo et al. 2002, Tang 2002). The technology did not gain acceptance until recently, however. Advantages of using SS as the drying medium as described in subsections 2.3.1 (Drying

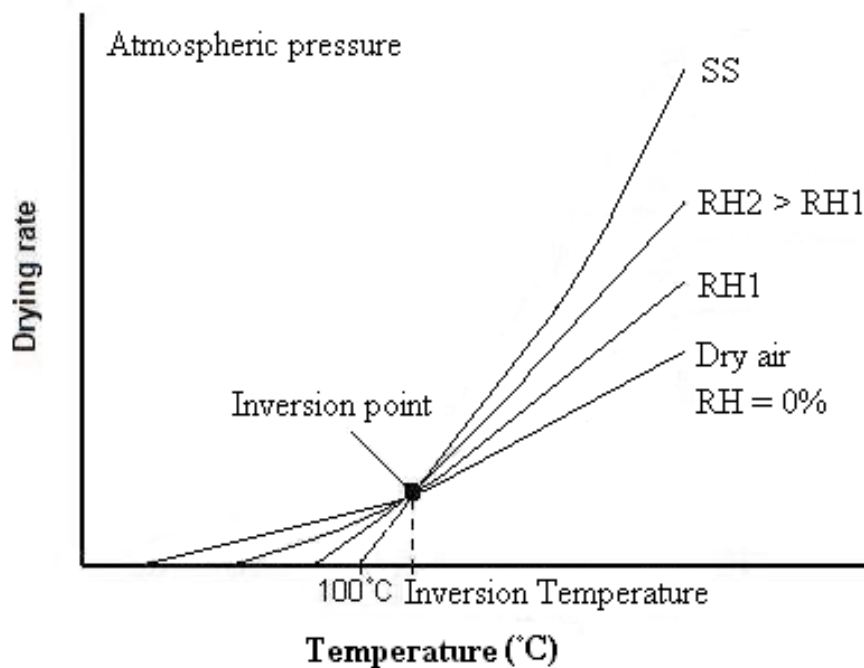
rates) through 2.3.5 (Benefits to foodstuffs) have been recently recognized by many researchers who began testing this new technology.

### **2.3.1 Drying rates**

The drying rate of a certain medium is defined as the removal of moisture from a material per unit time. Thus, drying rate is dependent on heat and mass transfer coefficients. For both SS and hot air drying, the temperature of the drying medium greatly affects the drying rate. The higher the drying temperature, the higher the drying rate can be obtained (Zielinska et al. 2009; Pronyk et al. 2004; Iyota et al. 2001a; Tang and Cenkowski 2000; Tang et al. 2000). Zielinska et al. (2009) reported that drying rates increased with an increase of SS temperature in drying of distillers' spent grains. With drying pressure at approximately 2 to 4 kPa above atmospheric pressure and a sample size of  $22 \pm 1$  g, a drying rate of 0.02 increased to 0.83 kg/min when steam temperature increased from 110 to 160 °C.

Iyota et al. (2001a) showed decreases in both hot air and SS drying time with increased drying temperatures when drying sliced raw potatoes. With hot air at 170 °C, drying times of 1500 and 2000 s were needed for sample moisture content to reach 0.2 and 0 kg/kg db (dry basis) respectively. With hot air at 240 °C, drying times of only 750 and 1100 s were needed to reach the same moisture contents of 0.2 and 0 kg/kg db respectively. In the case of SS, at 170 °C, the drying time was 2250 and 3000 s to reach moisture contents of 0.2 and 0 kg/kg db. At 240 °C, it took 750 and 1100 s to reach the same moisture contents respectively, which was the same as that with hot air drying.

Heat-transfer properties of SS are superior to those of air at the same temperature due to the absence of resistance to diffusion of the evaporated water in its own vapor (Kudra and Mujumdar 2002; Shibata and Mujumdar 1994) and the drying rate in the constant drying-rate period is dependent only on the heat-transfer rate (Hosseinalipour and Mujumdar 1995). Many authors (Shibata 2005; Pronyk et al. 2004; Tatemoto et al. 2001; van Deventer and Heijmans 2001; (Bond et al. 1994; Nishimura et.al 1994; Yoshida and Hyodo 1970;) cited by Iyota et al. 2001a) reported that the drying rate of SS was higher than that of hot air above a certain temperature known as an inversion point (Kudra and Mujumdar 2002). The inversion point occurs at a temperature at which the drying rates of both superheated heated steam and hot air drying are the same (Figure 3). At temperature below the inversion temperature, the heat-transfer coefficient of hot air is higher than that of SS. This means that the drying rate of hot air is greater than that of SS. At temperature above the inversion temperature, the heat-transfer coefficient of hot air becomes lower than that of SS, affecting the drying rate, *i.e.* the drying rate of hot air becomes lower than that of SS. Depending on various flow configurations and flow regimes (laminar versus turbulent boundary layer flow, impinging jet flow, and free convective flow over bodies of complex geometry), the inversion temperature can range from 160 to 200 °C (Kudra and Mujumdar 2002; Yoshida and Hyodo 1970, cited by Tatemoto et al. 2001).



**Figure 3. A comparison in change of the drying rate for superheated steam and air at atmospheric pressure and at various relative humidities corresponding to their drying temperatures. RH - relative humidity and SS - superheated steam (Adapted from Kudra and Mujumdar 2002).**

Studies have been done on the drying characteristics, drying rates, and the effect of SS drying on product quality in sugar-beet pulp, potatoes, and spent grain by Tang et al. (2000) and Asian noodles by Pronyk et al. (2004). It was observed that drying times decreased and drying rates increased when steam temperatures increased. Tang et al. (2000) reported that the decrease in the drying times and the increase in the drying rates for sugar-beet pulp were both higher when drying with SS compared to hot air. A 48% reduction in the drying time was obtained with an increase in temperature from 130 to 183 °C for SS and 34 % for hot air drying. Also, a 60 % reduction in the drying time was achieved for potatoes with an increase in temperature from 125 to 165 °C. These studies

showed agreement with the inversion phenomenon that drying rates of SS could exceed that of hot air drying when the drying temperature was above the inversion temperature.

Elustondo et al. (2002) developed a mathematical criterion to estimate the optimum working conditions in terms of initial drying rate for drying foodstuffs with SS. They showed that the drying rate always increased as either temperature or velocity increased. It was concluded that a faster initial drying rate resulted in a shorter overall drying time. Therefore, applying SS drying at temperature higher than the inversion temperature can result in shortened overall drying time compared to hot air drying, which increases the efficiency of a drying system.

### **2.3.2 Energy savings**

In the food industry, the most commonly used techniques for food preservation are thermal processing and dehydration. These techniques require high energy input, however. Heating contributes approximately 29 % of the total energy used in the food industry in the United States, which is equivalent to 7 % of the total electricity used by the manufacturing sector (Okos et al. 1998). In Canada, the food industry consumes about 10 % of the total energy used in the country (The Canadian Agricultural Energy End-Use Data Analysis Center, 1998). Conventional hot air drying is one of the most commonly used processes in food preservation, which requires significant amounts of energy. The process alone accounts for about 25 % of the energy used in the food industry in Canada (Drouet 1984, cited by Woods et al. 1994). Many authors (Shibata 2005; Pronyk et al. 2004; van Deventer and Heijmans 2001; Iyota et al. 2001b; Woods et al. 1994) claim that using SS as an alternative to hot air, the energy consumption for drying can be reduced



by 50 to 80 %. Superheated steam drying is always carried out in a closed circuit system (Wimmerstedt and Hager 1996). The SS is reusable by circulating and reheating. The steam generated (the amount of water evaporated) from the material is also circulated and can be used as the heat source or drying medium after being reheated in the system (Tang and Cenkowski 2000; Jensen 1995). The surplus steam only needs to be discharged when pressure exceeds the safety limit (Pronyk et al. 2004; van Deventer and Heijmans 2001; Tatemoto et al. 2001). Reusing allows energy to be recovered and greatly reduces the cost of heating (for generating steam). As mentioned in the previous section, the overall drying time can be decreased and the efficiency can be increased by increasing drying rates. A shorter overall drying time can also help saving the power expenditure of a plant and a higher efficiency can possibly contribute to a reduction in the equipment size and the capital cost. Such a potential in energy savings encourages studies and application of SS as the drying medium instead of hot air drying.

### **2.3.3 Reduced environmental impact**

In a closed circuit system, pollution problems can be avoided. Emission of odour, dust or other hazardous components is mostly prevented. For example, due to very limited ventilation gas stream, 90 % of dust emissions from drying of coal are reduced by SS compared to traditional driers (Woods et al. 1994). Furthermore, valuable organic liquids or products containing toxic compounds can be recovered. All these products can easily be collected in the condensate of the exhausted steam (Mujumdar and Huang 2007; Kudra and Mujumdar 2002; van Deventer and Heijmans 2001; Iyota et al. 2001b; Tatemoto et al. 2001; Tang and Cenkowski 2000, Jensen 1995; Woods et al. 1994).

#### **2.3.4 Oxygen-free environment**

In SS drying, the drying medium is only the SS with no oxygen present. Therefore, the risks of burning the material being dried or explosion hazards are excluded (Pronyk et al. 2008; Mujumdar and Huang 2007; Pronyk et al. 2004; Kudra and Mujumdar 2002; Iyota et al. 2001a; Iyota et al. 2001b; Tang and Cenkowski 2000; Jensen 1995; Woods et al. 1994). This is essentially important in drying fuels such as coal and bark. When dehydration of food takes place in an oxygen free environment, browning due to oxidative reaction is reduced in the final product (Jaya and Das 2003; Elustondo et al. 2001; Jensen 1995; Woods et al. 1994). Therefore, better quality of product can be obtained (Pronyk et al. 2004; van Deventer and Heijmans 2001; Woods et al. 1994).

#### **2.3.5 Benefits to foodstuffs**

According to Iyota et al. (2001a) and Jensen (1995), materials dried using SS, heat up quickly, which causes the effect of sterilization. Blanching, pasteurization, and deodorization of foodstuffs can also take place during the SS drying process (Pimpaporn et al. 2007; Devahastin and Suvarnakuta 2004; Pronyk et al. 2004; Kudra and Mujumdar 2002; van Deventer and Heijmans 2001).

#### **2.4 Disadvantages of using superheated steam**

There are some disadvantages in using SS as a drying medium. The industry lacks the proper equipment. Often the SS systems are more complicated and less economical compared to hot air systems (Pronyk et al. 2004; Tang and Cenkowski 2000; Tang et al.

2000). The number of major manufactures that provide SS dryers is very small (Kudra and Mujumdar 2002).

When drying with SS at atmospheric pressure, the steam temperature needs to be above 100 °C. The available technology is limited to drying a narrow range of products because high temperature of SS is not suitable for drying any temperature-sensitive materials. These are products such as foodstuffs and bio-products that melt or undergo glass transition (Pimpaporn et al. 2007; Devahastin et al. 2004; Markowski et al. 2003; Tang and Cenkowski 2000; Tang et al. 2000).

Moreover, the condensation and restoration periods are observed in the SS process but are absent in hot air drying. Compared to SS, hot air contains little or no moisture, therefore, the amount of condensation on the material practically does not exist. A material dried using SS goes through two more periods compared to material dried with hot air. Thus, the residence time of a sample in the dryer is longer (Kudra and Mujumdar 2002).

As SS passes through a chamber, some steam dries the sample and the excess steam is removed by a vacuum pump. Since the excess steam is not utilized in any other part of the system, the remaining steam condenses, eliminating the energy-saving potential. In contrast, more energy resource (such as cool water from a heat exchanger) is needed to cool down the surplus steam. In this situation, it is not economically and environmentally efficient.

## **2.5 Vacuum drying**

At atmospheric or higher pressure, both SS and hot-air drying result in high drying temperature. When dried at a pressure equal to or greater than atmospheric pressure, most food products are damaged, undergo glass transition, or melt because the melting points are lower than the drying temperature. Lowering the dryer operating pressure is one possible approach to preserve the product quality (Devahastin et al. 2004). Therefore, drying heat-sensitive materials such as foodstuffs, while maintaining quality, is possible when the operating pressure is lower than atmospheric pressure. This is also referred to as vacuum or low pressure drying.

### **2.5.1 Advantages of using vacuum drying**

Vacuum drying occurs when the removal of moisture from a material takes place under reduced pressure, which lowers the boiling point for water and provides a greater temperature difference between the heating medium and the material. This results in a higher drying rate and a more efficient heat recovery, making vacuum drying more beneficial compared to hot air drying. Sagar and Kumar (2010) and Jaya and Das (2003) reported that in vacuum drying, dehydration of food took place under low pressure and in the presence of very little or no oxygen. This also indicated that browning due to oxidative reaction was seldom present in the final product, which was advantageous. Air and water vapor present in food were expanded by vacuum and created a frothy or puffed structure. Improved heat and mass transfer were provided by the expanded structure of food offering a higher porosity. Therefore, a higher drying rate was achieved. Another advantage of vacuum drying is that it lowers the boiling point of water in a partial

vacuum, which causes vaporization of free water at temperatures below 100 °C and, at a rate close to that of high-temperature drying (Bousquet 2000, cited by Yamsaengsung 2008).

Amellal and Benamara (2008) dried date (*Phoenix dactylifera* L.) pulp cubes by reducing moisture content from about 14.0 to 6.5 % (db) under partial vacuum (20 kPa) at 60, 80 and 100 °C. No visual color change was observed, which indirectly agreed with Jaya and Das (2003), where no oxidation reaction was found under vacuum drying.

Devahastin et al. (2004) used carrot cubes as a heat-sensitive material and studied the drying kinetics of the dried product using low-pressure SS and vacuum drying. Several quality parameters of the dried product such as volume, shrinkage, apparent density, color, and rehydration behavior were evaluated. It was found that using SS under reduced pressure, product quality was superior to that of vacuum drying.

### **2.5.2 Disadvantages of using vacuum drying**

Compared to drying under atmospheric pressure, there are a few limitations of using vacuum drying. One limitation is the lower convective heat transfer due to low density of the drying medium at reduced pressure, which increases the drying time (Kudra and Mujumdar 2002).

Another disadvantage is the need for a vacuum pump, which increases the complexity of the system, capital cost and the amount of energy consumed (Thomkapanich et al. 2007).

### **2.5.3 Superheated steam vacuum drying**

Although there are many advantages of SS drying, the technology is not applicable to most foodstuffs or heat-sensitive material due to the high drying temperature and pressure (Wimmerstedt and Hager 1996). The problem can be solved by combining SS drying with vacuum drying, the effect of which is similar to hot air vacuum drying. Depending on more specific types of SS vacuum drying, not all of the advantages of SS drying and vacuum drying are inherent in SS vacuum drying. To be more precise, SS vacuum drying can be classified into two categories: continuous and intermittent.

### **2.5.4 Continuous superheated steam vacuum drying**

Superheated steam drying under continuous vacuum takes place when the operating pressure is lower than atmospheric pressure and is maintained at all times during drying. Similar to vacuum drying, when the processing environment is at a low pressure, steam density decreases and lowers its convective heat transfer capacity which greatly affects the drying rate and drying time (Devahastin et al. 2004; Elustondo et al. 2002).

Leeratanarak et al. (2006) investigated the drying kinetics and quality of potato chips dried under continuous low-pressure SS and hot air drying. They reported that low-pressure SS drying needed less drying time than hot air drying when temperature was above 80 °C. Also, in terms of browning index, better quality potato chips were obtained using low-pressure SS than hot air drying.

Suvarnakuta et al. (2005) conducted a study comparing the effects of continuous low pressure SS drying, hot air and vacuum drying on the drying kinetics of carrot. It was concluded that low-pressure SS drying generally required a longer drying time than vacuum drying. Unfortunately, the term “generally” was not specified. In terms of product quality, using low-pressure SS yielded better results than of hot air drying in terms of degradation of  $\beta$ -carotene.

### **2.5.5 Intermittent low pressure superheated steam drying**

Intermittent low pressure SS drying refers to drying using SS in an environment where atmospheric pressure is interchanged with lower-than-atmospheric pressure during the whole drying process. As previously mentioned, energy consumption of continuous vacuum drying is usually greater than that of atmospheric pressure drying due to continuous use of a vacuum pump. Therefore, the primary goal of using intermittent low pressure drying is to improve the energy efficiency. Thomkapanich et al. (2007) studied intermittent low-pressure SS and vacuum drying using banana as a heat-sensitive material. The study showed that higher drying rates were achieved by intermittent low pressure SS and vacuum drying compared to continuous low pressure SS and vacuum drying. This indirectly agreed with Devahastin et al. (2004) and Elustondo et al. (2002) who showed that lower processing pressure yields lower drying rate.

## **2.6 Superheated steam drying in different industries**

In recent years, SS drying has been getting much attention not only from the food processing industry, but also different industrial areas. The technology has been applied to drying lumber, paper, and fuel as described below.

### **2.6.1 Drying of lumber**

In the lumber industry, drying is an operation that substantially contributes to energy consumption in wood processing. It can consume 40 - 70 % of the total energy use in the industry (Rosen 1987, cited by Woods et al. 1994). The energy saving potential of using SS drying makes it attractive enough to be considered as a replacement for hot air drying. Moreover, wood color and brightness of the dried wood are two of the factors for determining the wood quality. Using low pressure SS instead of vacuum or hot air drying can enhance wood quality because the darkening of wood can be decreased by the reduced processing temperature and chemical staining can be avoided with absence of oxygen (Defo et al. 2004).

Pang and Pearson (2004) studied SS drying of softwood timber under both vacuum and absolute pressures from 20 to 600 kPa. It was reported that SS drying at pressures above atmospheric pressure increased the drying rate by about five times compared to the commonly used accelerated conventional temperature (ACT) drying, which dries with temperatures of 90 °C (dry-bulb) and 60 °C (wet-bulb). Superheated steam drying of softwood timber at atmospheric pressure resulted in a drying rate about 10 times faster than the ACT drying and five times faster than the normal high temperature drying.



Yamsaengsung and Sattho (2008) studied the effect of SS vacuum drying on the drying time and mechanical properties of rubberwood. Rubberwood boards were dried at 86.7 - 89.3 kPa vacuum pressure and temperatures of 60, 70, and 80 °C. The experimental results showed that the drying time was significantly reduced from 168 to 20 hours. Compared to conventional drying, SS vacuum drying produced higher quality rubberwood. The SS vacuum dried rubberwood had a 32 % increase in hardness, a 12 % increase in resistance to compression parallel to grain, and an 88 % increase in shear parallel to grain. All the increases in these properties were desirable.

### **2.6.2 Drying of paper**

Mujumdar (1981, cited by Kudra and Mujumdar 2002) first proposed the use of SS to dry paper. Aside from the advantages previously mentioned, SS drying can provide better quality of paper in terms of strength and visual properties (Cui et al. 1986 and David 1987, cited by Kudra and Mujumdar 2002). A study of SS drying of paper reported the significant enhancement in paper properties compared to paper dried using hot air (McCall and Douglas 2005). It was concluded that most of the paper composed of different types and ratio of pulps from the experiment yielded a better quality final product with improved properties such as higher tensile strength, toughness, brightness, and amount of bulk.

### **2.6.3 Drying of fuel**

Fuel such as peat, brown coal, and wood are dried to burn hotter and more vigorously than wet fuel. Typically, 25 % of the energy contained in the fuel is wasted to

evaporate water contained by wet fuel assuming for a wet wood fuel with 60 % moisture content. If the wet fuel contains only 40 % moisture, the energy waste drops by over one half to 11 % (Hulkkonen and Bruce 1998). Ireland and the former Soviet Union have used SS to dry peat for over 80 years. (Fagernas and Wilen 1988, cited by Kudra and Mujumdar 2002). In the early 1970s, Chalmers University of Technology in Sweden developed a SS dryer suitable for drying peat, pulp, and bark.

For cost-effective utilization, wet raw brown coal should be dried to about 5 to 10 % moisture content. Drying of coal containing 65 % moisture (wb) in a steam fluidized bed could yield a 15 % increase in the overall power plant efficiency (Kudra and Mujumdar 2002). Woods et al. (1994) stated that the grindability of coal could be improved and the sulphur content could be lowered up to 50 % using SS drying. They found that the constant-rate drying period was 6 to 7 times longer in the steam drying and the heat transfer rate was 1.7 to 2 times that in hot air. Clayton et al. (2007, cited by Kudra and Mujumdar 2002) studied drying of brown coal from 61 to 11 % moisture content (w.b.) using SS rotary dryer on a pilot scale system. They reported that the most significant effects on the removal of moisture were caused by feed rate and steam temperature.

All of the authors of the studies on SS drying of wood, paper, and fuel were consistent in their conclusions, SS drying is superior to hot air drying in terms of product quality and/or energy efficiency. Therefore, SS drying has the potential to be utilized in these industries.

## 2.7 Summary

Superheated steam drying is relatively young in the drying industry. Studies concerning drying foodstuff using SS in the processing area are limited. In addition, there are not many published works about SS under different conditions such as intermittent and continuous low pressure. Most of the research articles focus on comparing the effectiveness of SS drying to that of hot air drying under various conditions in terms of product quality, drying rate and drying time. Others tended to determine the optimum operating conditions of SS drying under different processing conditions. Most of these studies were solely quantitative research that was experimental based, only a limited amount of work showed theoretical analyses. More research (both quantitative and qualitative) is still needed to be done in the future.

A common problem with most of the articles included in this thesis is that authors seemed to refer to the results of only one or two specific author(s). For example, many authors did not specify the exact value or range of the occurrence of the inversion temperature and some of them referred to the work of Yoshida and Hyodo from 1970. In the future, more research should be done on the inversion temperature in order to have a significant number of research results with which results can be compared.

All of the research studies showed consistency in their experimental results. Common parameters such as drying rate, drying time, and product quality (shrinkage, color, rehydration ability, hardness, toughness, crispness, and ascorbic acid retention for foodstuffs; brightness, shear resistance, compression resistance, modulus of rupture, modulus of elasticity, and hardness for wood; color, brightness, density, and shear

resistance for paper) were evaluated, compared and commented on. A common comment among the studies was that SS drying could be a potential replacement for hot air drying for a number of products.

## **2.8 Gaps identified in current research**

There are two points found to be contradictory. Some authors stated that products that were air dried experienced case-hardening, which made the surface of the product water-impermeable but products that were dried by SS did not form such shells (Leeratanarak et al. 2005; Kudra and Mujumdar 2002). Other authors reported that case-hardening was observed from their experiments (Kerdpi boon and Devahastin 2007; Pronyk et al. 2004; Iyota et al. 2001b; Tang and Cenkowski 2000) using SS drying or both hot air and SS drying. From the results presented, it is consistent that case-hardening is observed on air dried products. Also, case-hardening occurs on products dried using SS but comparatively, it is more likely to happen on air dried products. Furthermore, different authors had different dried materials. Case-hardening can be material dependent. These can explain why some authors observed case-hardening on air dried products but not on those dried using SS.

The second contradictory point is related to the drying rate. Some authors (Leeratanarak 2006; Jaya and Das 2003) claimed that the drying rate at low pressure is higher, while other authors (Kudra and Mujumdar 2002) stated that it is lower. According to Elustondo et al. (2002) and Wimmerstedt (1995), heat transfer capacity is diminished because of the reduced steam density at low pressure. On the other hand, heat transfer driving force (the difference between SS temperature and material surface temperature) is

reduced by the increased water equilibrium temperature due to higher pressure. In both cases the authors can be correct although they are not in agreement with each other. Since the literature pertaining to this subject is limited, more research is needed to be done to confirm both cases.

All studies agreed that SS drying is better than hot air drying only under certain conditions. These conditions include: higher temperature above the inversion temperature, higher velocity of flow, and intermittency of low processing pressure. None of these were reported in numerical values and only a very limited amount of paper has commented on them. Gaps have also been identified with regards to the experimental materials. Most studies regarding food processing have only used materials such as potatoes, carrots, apples and bananas to test the SS system, however not many other varieties of food have been tested. Moreover, the physical quality of food product was the main focus with no chemical or nutritional analysis. Whenever SS was concluded to have higher energy efficiency than that of hot air, no actual calculations of energy consumption were presented. Only qualitative results were given in most cases

## 3 MATERIALS AND METHODS

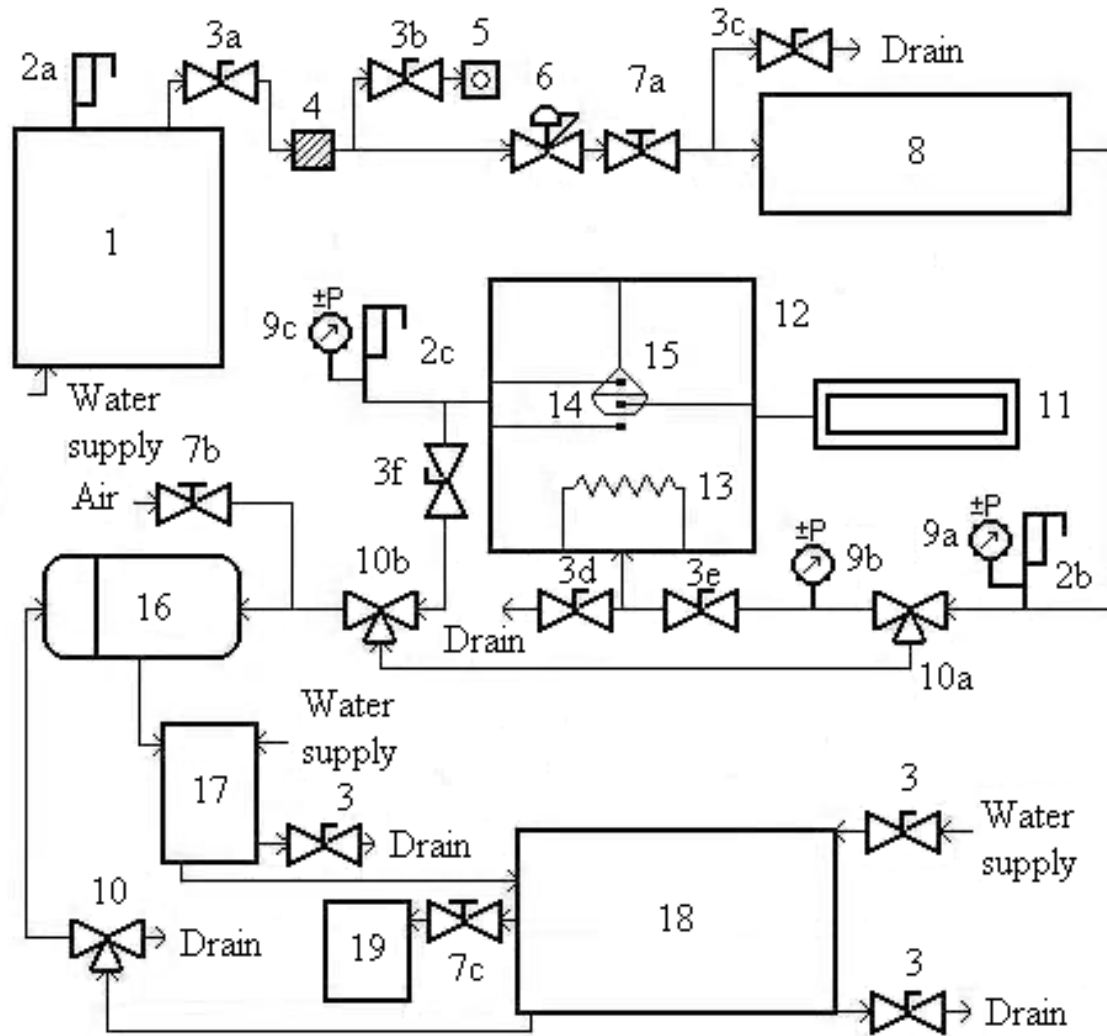
### 3.1 Material

Whole stillage used in the SS drying experiments was obtained from a distillery (Mohawk Canada Limited, a division of Husky Oil Limited) in Minnedosa, Manitoba. This plant processes wheat and corn into bio-fuel. Samples obtained from the Minnedosa plant were a blend of 75 % corn and 25 % wheat in solid content. Samples were obtained by separating the whole stillage into solid distillers' spent grain (DSG), paste, and liquid fractions containing soluble material using a Sorvall RC-3 centrifuge (Thermo Scientific Co., Asheville, NC). The centrifuge operated at a relative centrifugal force of  $790 \times g$ , with four 1000 mL containers rotating for 10 minutes at 2200 rpm, on a radius of 0.146 m. After the centrifugation, solid and liquid fractions were separated by manually scooping out the fractions from the containers. After separation, the solid fraction was bagged and stored at  $-15\text{ }^{\circ}\text{C}$ . Frozen samples were thawed at room temperature prior to running experiments. Only solid DSG (fraction with highest density) was used in the experiments. The air-oven drying method (AACC, 2000) was used to determine the moisture content of the solid DSG, which was found to be  $77.6 \pm 0.9\%$  wb.

### 3.2 Experimental set-up

The SS system used in the experiments was developed in the Department of Biosystems Engineering at the University of Manitoba. Figure 4 shows the flow and components of the low-pressure SS system.

The main components of the low-pressure SS system included (1) an electric boiler, (2) three safety relief valves, (8) an electric superheater, (6) a pressure regulator, (12) a drying chamber, (13) an electric heating tape, (16) a vacuum pump, (17) a heat exchanger, and (18) a reservoir. Other accessory units included steam conveying piping, (4) a strainer, different types of valves including (3) ball, (7) globe, and (10) three-way valves, (5) a vacuum breaker, (9) three vacuum-to-positive pressure gauges, (11) a data acquisition system, (14) seven thermocouples, and (19) an overflow reservoir. Details of components are discussed in the following sections.



**Figure 4-1. A flow diagram of the low-pressure superheated steam system.** 1 – electric boiler, 2(a,b,c) – safety relief valves, 3(a,b,c,d,e,f) – ball valves, 4 – strainer, 5 – vacuum breaker, 6 – pressure regulator, 7(a,b,c) – globe valves, 8 – superheater, 9(a,b,c) – vacuum-to-positive pressure gauges, 10(a,b) – three-way valves, 11 – data acquisition system, 12 – drying chamber, 13 – electric heating tape, 14 – thermocouples, 15 – sample, 16 – vacuum pump, 17 – heat exchanger, 18 – reservoir, 19 - overflow reservoir.



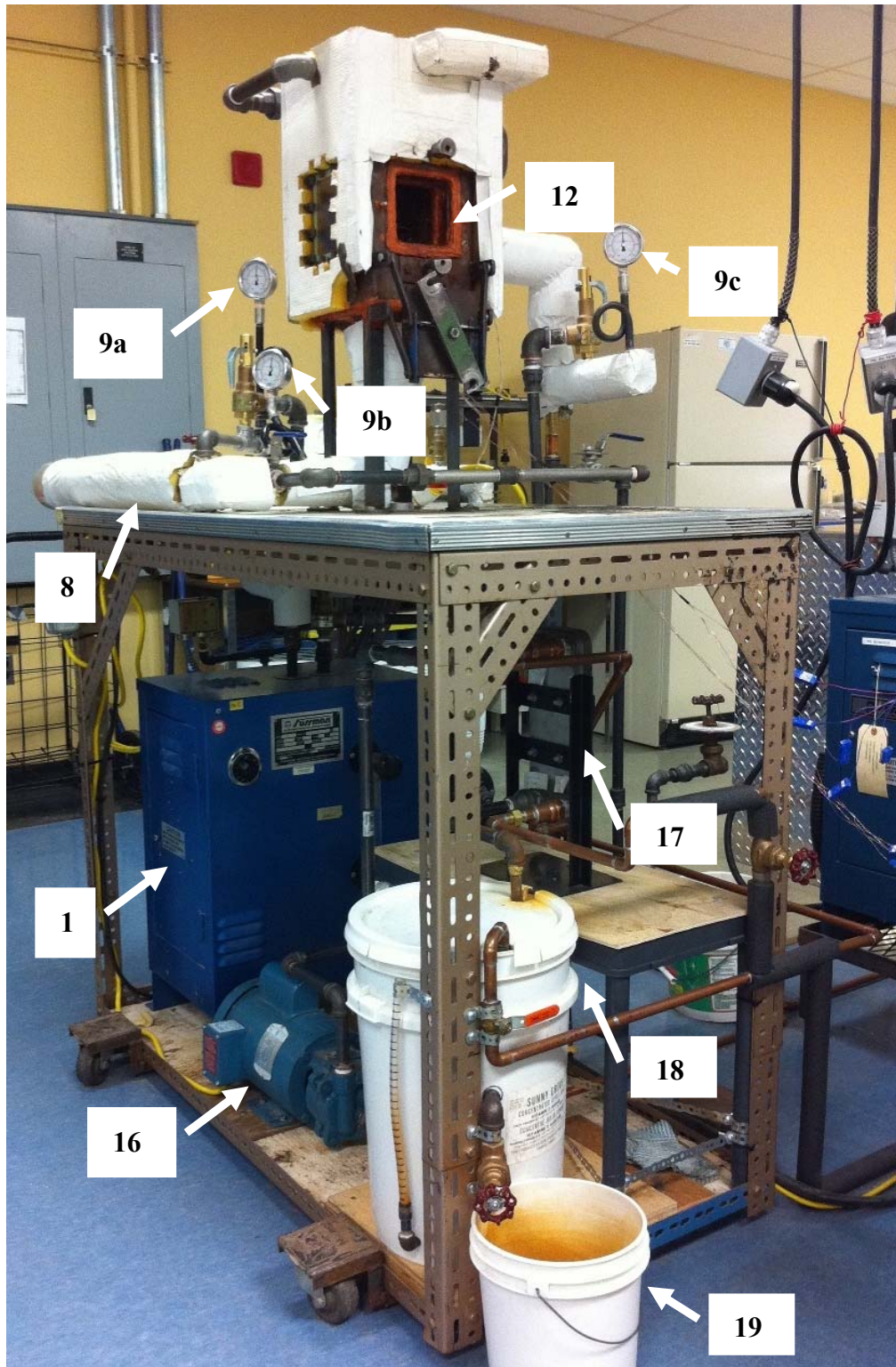


Figure 4-2. Custom designed low pressure superheated steam processing unit (refer to Figure 4-1).

### **3.2.1 Electric boiler**

The 9 kw boiler (#1, Figure 4) (SUSSMAN Electric Steam Generators, model MB9L, Long Island City, NY, USA) generates steam at a rate of 12.3 kg/h. It requires 3 Phase, 208V at 50A. The maximum allowable working pressure is 689 kPa, which is sufficiently above the maximum system operating pressure.

### **3.2.2 Safety relief valve**

Three safety relief valves (#2a, #2b, and #2c, Figure 4) are used in the system, the one with the highest (689 kPa) pressure limit (Kunkle Valve, Model B021 DC01, Edmonton, AB, Canada) was located at the beginning of the system where the boiler (#2a) is, one with a pressure limit of 207 kPa (Apollo Valves, Model 19KDCL30, Matthews, NC, USA) is situated right after the superheater (#2b) and one with a pressure limit of 103kPa (Apollo Valves, Model 19KDCL15, Matthews, NC, USA) is located right after the drying chamber (#2c). Although there is a switch on the boiler to control the maximum steam pressure, a safety relief valve (#2a) is needed in case of any malfunction of the control. As the superheater (#8) is another heating source, there is a potential pressure build up. A safety relief valve is needed to release pressure if the SS pressure is too high. A third safety relief valve (#2c) is placed at the outlet of the drying chamber, because the drying chamber becomes a closed volume when the pipeline is rerouted to by-pass it. Pressure could build up as there is no opening. All three safety relief valves are necessary as they protect both system and surroundings from explosion due to a possible pressure build up.

### **3.2.3 Vacuum breaker**

A vacuum breaker (#5, Figure 4) (Watson McDaniel Company, model WVBSS, Pottstown, PA, USA) is positioned at the pipeline close to the electric boiler to allow air to enter the system in order to “break the vacuum” caused by the condensing of steam or draining of liquid after the system is shut down. Elimination of the vacuum is necessary to allow for proper drainage of liquid from the system and protect the processing equipment. As the vacuum breaker functions like a simple check valve, it closes off tightly when steam or water tries to escape and it only draws air into the system when the system experiences vacuum. Therefore, the vacuum breaker has to be installed in a vertical position to function properly. Initially vacuum created by the vacuum pump has not affected the performance of the vacuum breaker (when it was new) as the vacuum pump is only turned on when the electric boiler is in operation. The pressure built up in the pipeline near the boiler is high enough to offset the vacuum created by the pump at the position where the vacuum breaker is. However, the vacuum breaker developed air seepage over time. The seepage problem will be further discussed in Section 4.1.3 Sudden temperature drop. The vacuum breaker has a maximum operating pressure and temperature of 300 psig or 2068 kPa and 400 °C, which are in excess of the system requirements.

### **3.2.4 Pressure regulator**

A pressure regulator (#6, Figure 4) (Fisher Controls International Inc., Type 95L NPT, Marshalltown, IA, USA) is implemented for three reasons. First, in spite of the boiler pressure control and the safety relief valve, the pressure regulator further reduces

the pressure of steam entering the system serving as a safety mechanism. Secondly, lowering the pressure helps decrease the flow rate which gives steam a longer time to absorb heat when passing through the superheater, and enables a steady flow of steam through the pipeline and the drying chamber. More importantly, saturated steam produced by the steam generator at 275 kPa, is reduced to a pressure of 103 kPa (slightly above atmospheric pressure) by the pressure regulator, which contributes in superheated steam generation.

### **3.2.5 Electric superheater**

The electric superheater (#8, Figure 4) (Accutherm Inc., Monroe City, MO, USA) adds sensible heat to the saturated steam generated from the boiler, increasing the temperature of the steam, resulting in SS generation. The superheater has a 1.5 kW single phase, 240 V heating element, and a thermostat for regulating temperature ranged between 79 and 288 °C.

### **3.2.6 Pressure gauges**

Three vacuum-to-positive pressure gauges (#9a, #9b, and #9c, Figure 4) (Winters Instruments, Type 233.53, Toronto, ON, Canada) with a range between -100 and 100 kPa (gauge pressure) were implemented in the system. One (#9a) was placed at the pipeline after the superheater acting as a safety mechanism as a monitoring device. Since steam pressure is controlled by the pressure regulator, an inlet steam pressure of approximately 103 kPa is expected. The 100 kPa (gauge pressure) upper pressure limit is sufficient to measure the increase in steam pressure by the superheater. The lower pressure limit

of -100 kPa (gauge pressure) is well below the lowest designed pressure, which is about -25 kPa (gauge pressure). Another two vacuum-to-positive pressure gauges (#9b and #9c) are positioned before and after the drying chamber. The chamber pressure can be obtained by finding the average pressure level of the two gauges.

### **3.2.7 Electric heating tape**

A 144 W electric heating tape (Omega Engineering Inc., HTWAT051-004, Laval, QC, Canada) was wrapped around the pipes connecting the superheater and the drying chamber to keep the steam above the desired superheat temperature. The attached adjustable thermostat controlled the tape temperature from 10 to 218 °C. The maximum exposure temperature was 232 °C, which was well above the maximum system operating temperature. Even though the pipe supplying steam wrapped with heating tape was also insulated, the heating tape ensured that the temperature of superheated steam did not decrease when it passed through the supply pipes before reaching the drying chamber. Thus, the possibility of condensation due to heat loss was minimized.

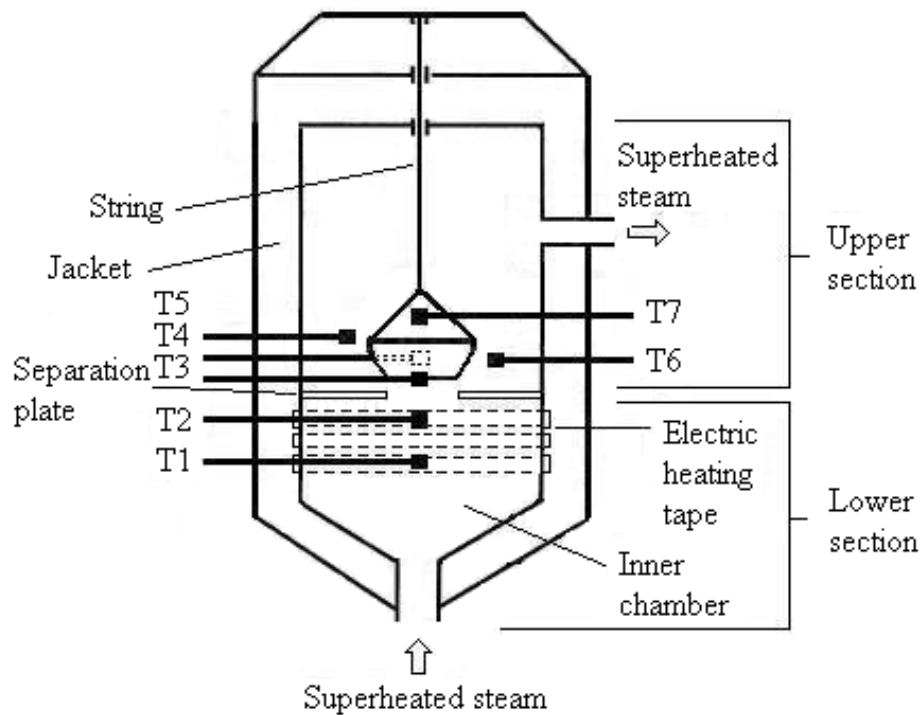
### **3.2.8 Drying chamber**

The drying chamber (#12, Figure 4) consists of an inner chamber where the sample is placed, and a surrounding air jacket (Figure 5). There is a separation plate dividing the inner chamber into two sections. The lower section of the chamber is where the SS enters through the piping. The separation plate is to partially block the SS from coming in contact with the sample before filling the lower section. The separation had a circular opening with a diameter of 68 mm, which allows for the passage of SS. By filling

the lower section with SS, the temperature loss of SS is reduced before contacting the sample, thus resulting in a more uniform temperature distribution in the chamber. An electric heating tape is also placed inside the air jacket and wrapped around the walls of the lower section of the inner chamber. The heating tape warms up the whole chamber prior to experiments, reducing heat loss from SS, reducing the potential for condensation during experiments. The warmed air jacket acts as an insulation of the inner chamber, slowing down heat transfer from inner chamber to the atmosphere. A glass fibre pipe wrap is also used from the piping of boiler to the outlet of the drying chamber as an external insulation to prevent heat loss from the system. All components after the drying chamber outlet are not insulated as the steam after that point is needed to be cooled to prevent high temperature steam entering the vacuum pump, which might be overheated. The sample container is suspended in the upper section of the chamber 15 mm above the circular opening where it is close to the heating tape in the lower section of the chamber. The upper section has a cross-sectional area of  $0.01472 \text{ m}^2$  and is accessible through a chamber door for sample placement.

### **3.2.9 Thermocouples**

Seven thermocouples (T1 to T7, Figure 5) are inserted into the drying chamber, one to measure sample's temperature and the remaining six to measure the temperature at various points within the chamber (Figure 5). The T-type thermocouples are used because they are suited for measurements up to  $350 \text{ }^\circ\text{C}$ , which is well above the maximum drying temperature of the experiments.



**Figure 5. Drying chamber.**

### 3.2.10 Three-way valve

Three-way valves (#10a, and #10b, Figure 4) are used to reroute the flow of the SS. When the sample needs to be removed in the middle or after the drying process, it could be hazardous since the SS is still flowing and the chamber temperature is still very high. Use of three-way valves enables the flow to bypass the chamber, which lowers the risk of the operator getting burned and avoids the escape of SS through the chamber door.

### 3.2.11 Data acquisition system

A data acquisition system (#11, Figure 4) (Agilent Technology Canada Inc., Agilent 34970A, Mississauga , ON, Canada) was used to monitor the temperatures of the

sample and the inside of the chamber. Temperature values were monitored with seven thermocouples. Temperatures were recorded every second.

### **3.2.12 Vacuum pump**

The liquid ring vacuum pump (#16, Figure 4) (SIHI Pumps Ltd, Model LEM 20, Guelph, ON, Canada) creates suction using its eccentrically mounted impeller, which forms different sizes of cells within the casing. The pump requires a continuous supply of cooling liquid (tap water) to absorb heat generated and collected during the operation to safeguard the pump against damage from overheating. Steam from the chamber is introduced into the cells and mixed with cold (tap) water, which enters the pump on the suction side and is discharged. To eliminate the cooling liquid make-up and drain needs, a complete recirculation was implemented. When the mixture is discharged through the outlet of the pump, it is still at a relatively high temperature. Therefore, a heat exchanger is installed at the outlet of the pump, thus cooling the mixture. After cooling, the mixture is recirculated back to the inlet of the cooling liquid port of the pump. Due to the implementation of complete recirculation, the amount of cooled water recirculated back to the pump slowly increases. As SS cools and condenses into water, the total volume of cooling water continuously increases. Therefore, drainage of excess cooled water is needed occasionally. The excess amount of water is the rate of SS flow, which is identified by condensation.



### **3.2.13 Heat exchanger**

The heat exchanger (#17, Figure 4) (GEA PHE Systems, Model MPN5X12-8, Newmarket, ON, Canada) has eight pieces of nickel brazed, 254SMO stainless alloy plates, giving high efficiency heat transfer for applications up to 757 liters per minute, which is much greater than the maximum SS flow rate. The heat exchanger is installed in a vertical position to ensure proper drainage of condensate.

### **3.2.14 Reservoir**

The reservoir (#18, Figure 4) is used as storage of cooling liquid for the vacuum pump. As mentioned previously in section 3.2.12 Vacuum pump, the complete recirculating system causes additional water to collect from condensate, which enables the determination of the SS flow rate. This is measured by looking at the amount of water gathered from the overflow reservoir. The overflow condensate flows through an extra globe valve (#7c), installed at the mid-level of the reservoir and is collected in a separate reservoir (#19). The location of the extra globe valve allows the excess water to discharge without substantially depleting the cool water. When condensation occurs, the water level rises and the excess water flows through the extra globe valve (#19). The amount of water collected from the overflow reservoir is used to calculate the SS flow rate by looking at the volume of water obtained per unit time.

### **3.3 Experimental procedure**

#### **3.3.1 Sample preparation**

A semi-spherical metal mesh was used as the sample container. The metal mesh had a porous surface, which allowed SS to pass through the sample. Each sample of approximately seven grams of solid DSG was placed in the container and evenly spread out in a thin layer to cover the base and up the sides of the container, having a thickness of approximately two to three millimeters. The sample was pierced several times with a wire along the sides. The holes created by piercing, enabled the steam to flow through the pores, allowing for more thorough drying. The bottom of the grain sample was left intact to allow a thermocouple to penetrate the sample for a more accurate reading of the sample temperature throughout the drying process. The placement of the sample onto the thermocouple allowed for adequate adjustments to maintain the drying temperature of the grain at the desired pressure. A metal screen was put on top of the sample and locked in place with a wire. The metal screen helped to prevent the sample from being blown away by the flow of SS and especially the sudden increase of flow when the vacuum pump was turned on, while allowing SS to flow through the sample easily.

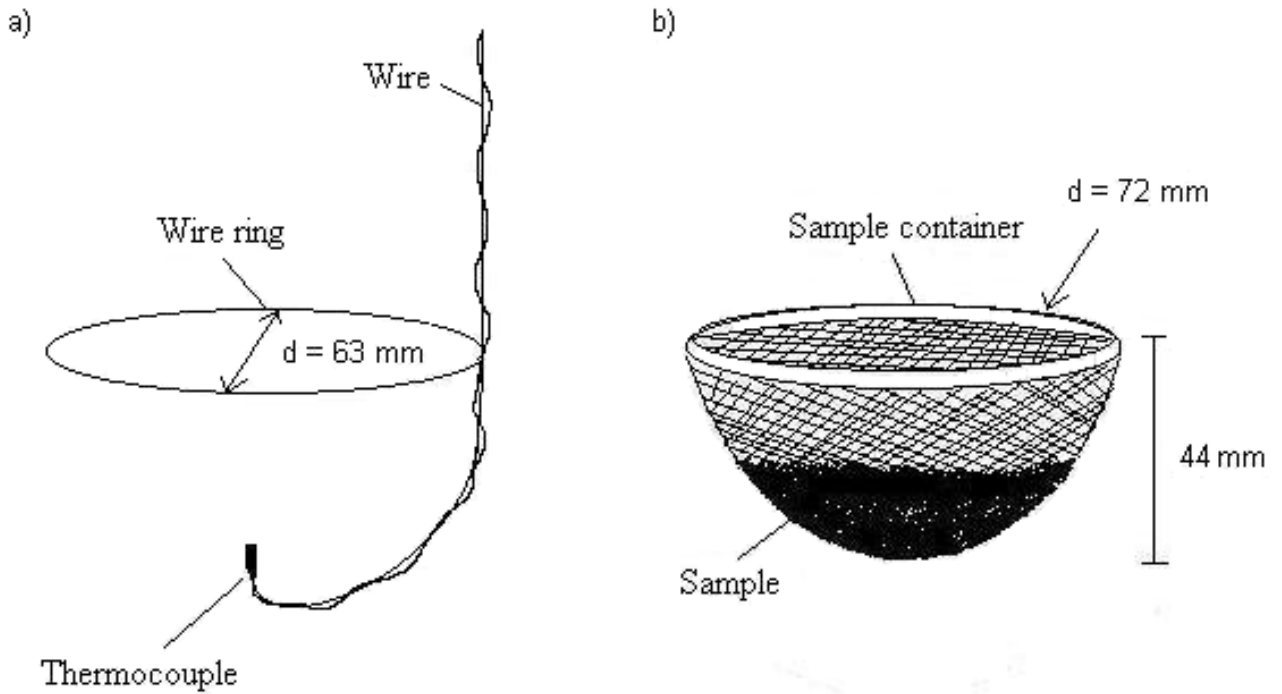
#### **3.3.2 System preparation**

Prior to the beginning of each experiment, the boiler (#1, Figure 4) was turned on as it took approximately 30 minutes to generate enough steam to supply a steady flow. The valve connecting to the vacuum breaker was closed. This valve was only opened before the boiler was turned off at the end of the experiment. Before inserting a sample,

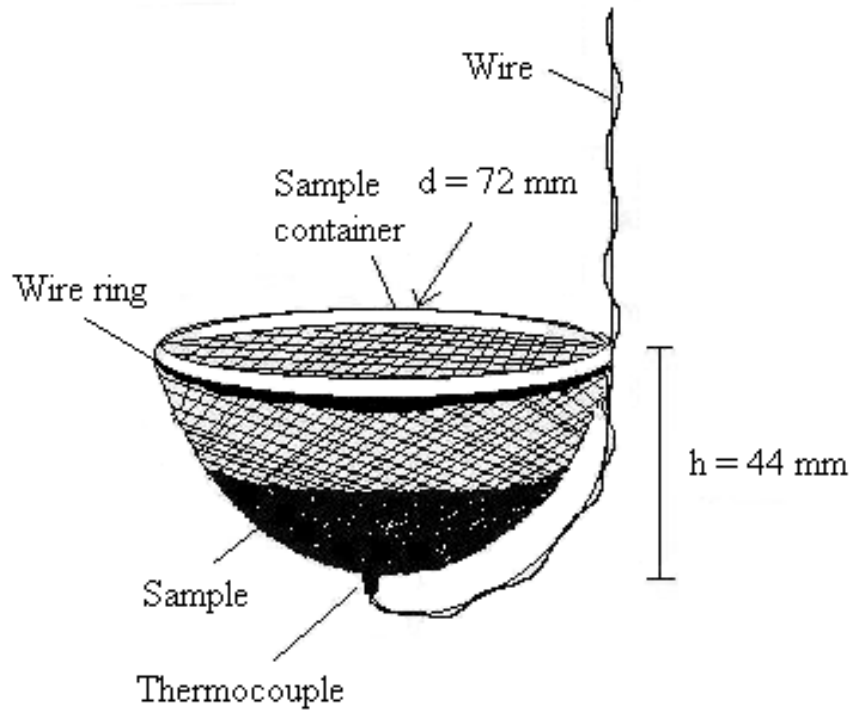
an electric heating tape (#13) was turned on to warm up the inner chamber. Then, residual water was removed from the pipeline leading to the superheater and the chamber by opening the valves installed below the superheater and the chamber (#3c and #3d respectively). The removal of the residual water from the previous experiment prevented the newly produced steam from condensing on cold water which would delay the production of SS, increasing drying time. The superheater (#8) was then turned on to warm up the air inside the superheater and piping. As soon as the boiler reached its maximum steam capacity (when boiler pressure was at the pre-set maximum limit), the valve connecting the boiler and the superheater (referred to as steam-flow valve) (#7a) was opened by turning it by approximately 30 °. Pressurized steam from the boiler was directed through the pressure regulator (#6) and then to the superheater, which was at a lower pressure than the boiler. Steam was superheated as it passed through the superheater. Before inserting a sample, the vacuum pump (#16) was switched on and SS was allowed to flow through chamber (#12) and warm it up. This reduced condensation of steam on the sample. When chamber temperature reached the desired drying temperature, the two three-way valves (#10a and #10b) were adjusted to bypass the SS. While the SS bypassed the drying chamber, the chamber door was opened and sample was placed inside the chamber.

The sample container was held securely in the chamber by placing it onto a thin wire hook, which was attached to the top of the chamber, and into a wire ring where a thermocouple would pierce the bottom of the sample (Figure 6 and 7). Suspending the sample exposed fully its surface to heat and water transfer. After the sample was placed

inside the chamber, the chamber door was closed. The two three-way valves (#10a and #10b) were adjusted to re-direct flow through the chamber and the drying process begun.



**Figure 6. A wire ring acting as a sample container holder (a) and sample container with a sample inside (b)**



**Figure 7. A sample container sitting in a wire ring**

### 3.3.3 Drying process

After the vacuum pump was turned on and started evacuating SS, SS began to flow in the chamber and through the suspended sample, beginning the drying process. To achieve a desired operating pressure, a valve installed close to the end of the inlet piping of the vacuum pump (referred to as air-valve) was opened and adjusted. Opening the air-valve caused the vacuum pump to draw atmospheric air into the outgoing SS flow, relieving negative pressure within the drying chamber. Alternatively, the air-valve could be closed to increase vacuum in the chamber. Therefore, pressure was controlled by adjusting the air-valve. The temperature of the system was controlled by adjusting the steam-flow valve, which regulated the amount of steam going to the superheater. Increasing the flow of steam going into the superheater created more SS and thus provided a higher temperature in the drying chamber. When adjusting the steam-flow valve, the pressure of the system was affected by a small amount, the air-valve was adjusted to compensate the loss/gain of pressure accordingly. Temperatures of the sample and inner chamber were monitored and recorded during the entire drying process every second using a data acquisition system. The total drying time was set to a maximum of 160 minutes depending on the pressure of the drying chamber and the temperature of steam. The steam temperature during drying ranged from 95 to 115 °C. The pressure held within the drying chamber was either -25 or -20 kPa. The SS velocity passing through the drying chamber ranged from 0.100 to 0.289 m/s.

### 3.3.4 Sample mass change measurement

Two methods of measuring sample mass changes were used. During the earlier stage of the experiment, the multiple-experiment method was used. In order to eliminate possible error and obtain a more accurate result in plotting the sample moisture content versus drying time curve, the sample in every experiment stayed in the chamber for the whole drying process, without being taken out and weighted. This minimized the disturbance to the sample, allowing constant drying conditions during the entire drying process. For example, to construct a sample moisture content versus a 160-minute drying time graph with 5 intervals at 20, 40, 80, 120, and 160 minutes, 5 different experiments were conducted. Each interval represented an individual experiment, therefore, the time needed for one graph was the sum of the intervals, which was  $20 + 40 + 80 + 120 + 160 = 320$  min.

Due to the amount of time needed for the multiple-experiment method, a different method – single-experiment method was used to construct the drying characteristic graphs. Instead of conducting one experiment for one single point on the graph, the sample was taken out and weighed during the experiment. With this method, every experiment went through a total drying time of 160 minutes. The time intervals on the graph were 20, 40, 80, 120, and 160 minutes, meaning that the sample were taken out during these intervals, and was placed back into the chamber to continue drying, until the 160-minute period was over. Using this method of measurement, only one experiment was needed to construct one complete graph, which was more time efficient. Using the single-experiment method, the mass of sample was measured during the drying process every 20 to 40 minutes. To do so, SS was redirected to bypass the drying chamber using

the three-way valves. Then, the sample was immediately taken out from the chamber and weighed. After, the sample was placed back into the chamber and SS was allowed to flow through to continue drying. The difference between the mass measured corresponding to the specific time and the complete dried mass was used to determine the moisture content of the sample. More detail of these two methods will be further discussed in section 4.2

Drying experiments under vacuum.

## **4 RESULTS AND DISCUSSION**

### **4.1 Preliminary testing of the equipment**

#### **4.1.1 Excessive suction**

While testing the system, it was observed that the vacuum pump was too powerful, creating negative pressure reaching as low as -40 kPa. The resulting suction pulled any saturated steam, generated by the boiler, through the superheater at an excessive pace, hindering the transition to superheated steam. In addition, water within the water level indicator oscillated rapidly and the pressure within the boiler struggled to rise, suggesting that the suction from the vacuum pump was sufficient to pull water directly from the boiler rather than produce an adequate flow of steam. An experiment was conducted to verify this. A droplet of water was placed in the drying chamber, and it was observed that the droplet was pulled to the outlet by the vacuum pump. Therefore, the water was only able to absorb a nominal amount of heat from both the boiler and superheater and did not turn into steam.

Since the vacuum pump did not have a switch to control the level of suction, one possible method to reduce the suction was to create a variable suction inlet. A valve (as discussed in section 3.3.3 Drying process) was installed downstream of the inlet piping. The level of suction could then be controlled by opening and adjusting the valve accordingly. After the installation of the air-valve, the system was tested. While the system was held at the desired operating pressure (-25 to -20 kPa), the water level within

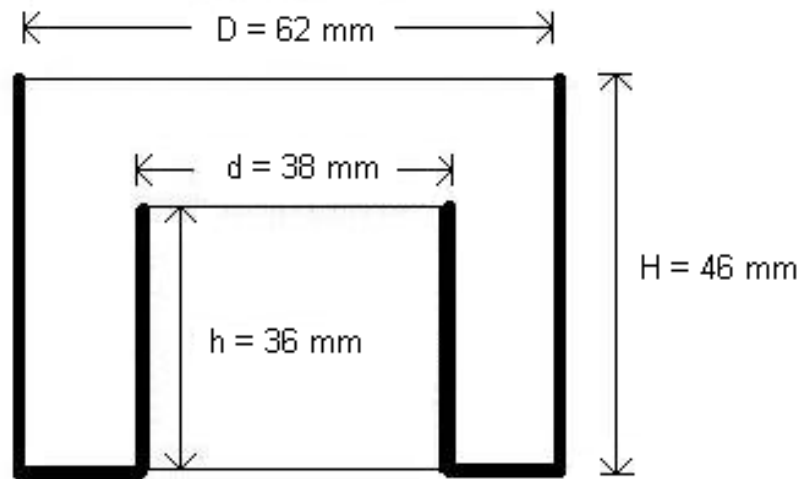


the boiler remained stable and the internal pressure increased, indicating that the boiler was producing steam.

#### **4.1.2 Lower-than-theoretical boiling point of water**

To ensure drying occurred in the drying chamber at the predicted temperature, several experiments were conducted to determine the practical boiling point of water. The experiment setup and the procedures were exactly the same as described in section 3.3.2 System preparation and 3.3.3 Drying process, except that water, rather than spent grain, was used as the sample. A different container (Figure 8) was fabricated to hold the water sample, enabling drying while monitoring temperature. The container had a thickness of 0.6 mm, it was made out of a 46 mm high cylindrical aluminum drying container with an inner diameter of 62 mm. The container was completely open on one end (top), fully closed on the other (bottom) and was suspended in the drying chamber. Due to the large surface area of the bottom of the container, there was a possibility that the water within the container would have minimal contact with the superheated steam. As steam was allowed to flow through the chamber, the bottom of the drying container would act as a barrier, diverting steam along the sides. In addition, the suction from the vacuum pump would continuously pull steam out of the chamber, inhibiting the sample from interacting directly with the steam. As a means to increase the interaction between the water sample and superheated steam, the drying container was altered. A hole was cut out of the bottom center of the container and a smaller cylindrical piece without a top or bottom was attached within the container (Figure 8). This enabled the steam to flow through the container and spread throughout the chamber more evenly, increasing the interaction

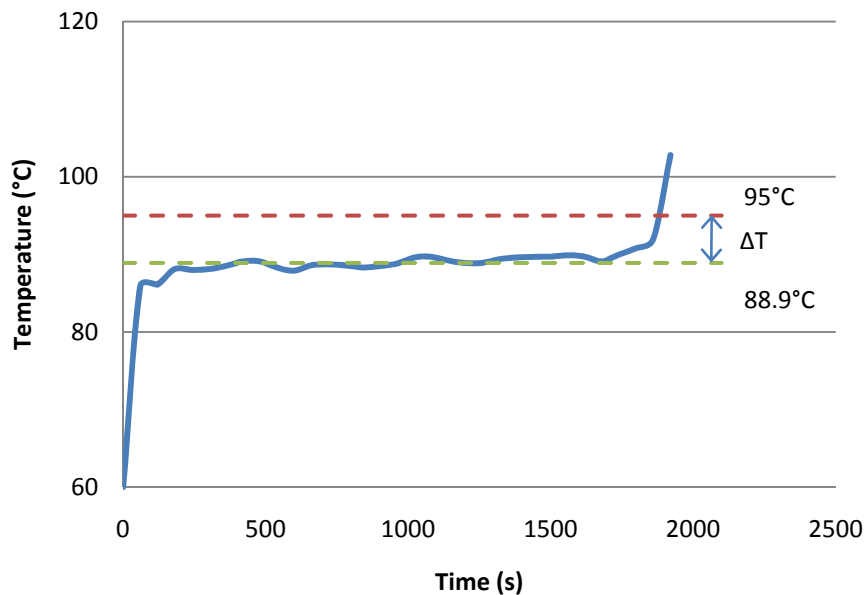
between the sample and steam by increasing the surface contact area. The inner cylinder had a thickness of 2.1 mm and an outer diameter of 38 mm and height of 36 mm. In addition, a mesh grate was fitted over the inlet to the drying chamber, reducing the flow of steam and serving as a resting place for the container, eliminating the need to suspend the sample. The large porosity of the mesh grate allowed steam to flow through the chamber at a slower rate and with a more even distribution. Superheated steam was then able to accumulate and circulate within the chamber, drying the water sample directly.



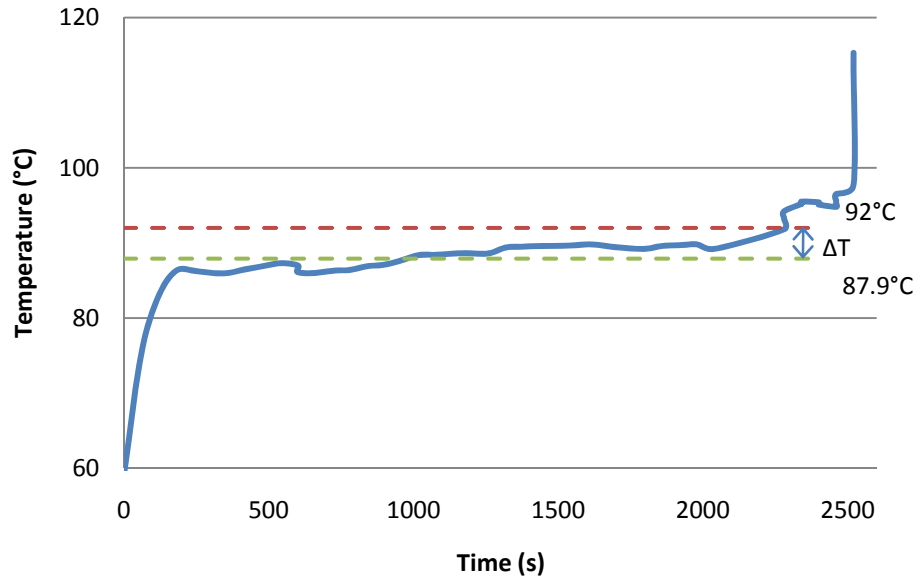
**Figure 8. Cross-section of the sample container for the boiling point confirmation experiment.**

When SS was introduced into the chamber, heat from the SS was transferred to the sample causing the water temperature to rise. The water temperature increased continuously, until the beginning of phase change - turning into steam. During the phase change, the water temperature stayed constant. Once all of the water turned into steam, the temperature of the steam increased, provided that SS was supplied continuously. The

boiling point could be found by plotting water temperature versus time. The horizontal part of the temperature characteristic indicated the boiling temperature in the chamber at the specific pressure. At a pressure of -20 kPa, drying (evaporation of water) occurred at an average temperature of 88.9 °C, while the theoretical boiling point for the same pressure was 95 °C. At a pressure of -25 kPa, drying occurred at an average of 87.9 °C, while the theoretical boiling point at that pressure was 92 °C. Figure 9 and 10 show the water temperature history in the chamber indicating the boiling point difference ( $\Delta T$ ). Therefore, it was concluded that the drying chamber was unable to maintain the SS conditions which at the end lowered the boiling points at the corresponding pressures. This was probably due to air seepage development.



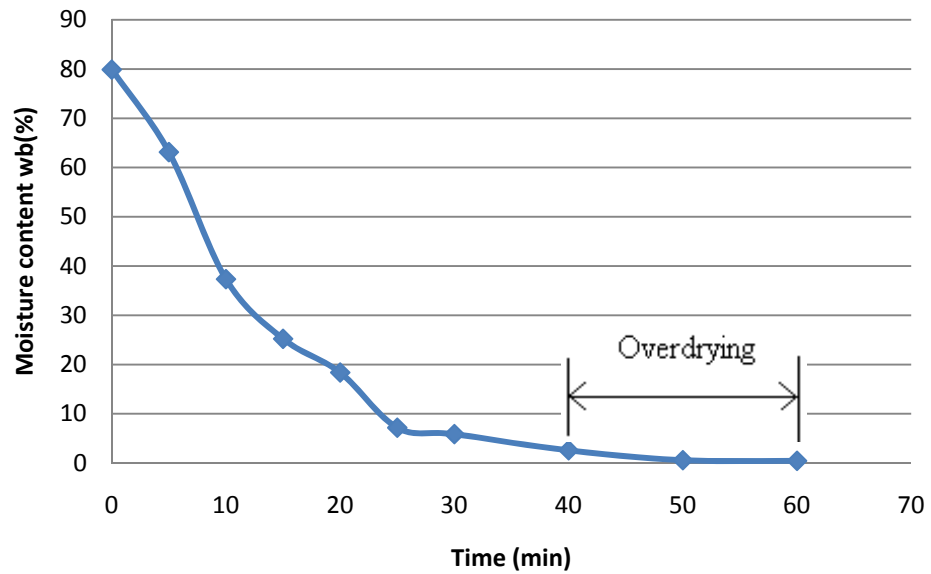
**Figure 9. Temperature change of water versus time for boiling point confirmation experiment under -20 kPa with an average evaporation temperature of 88.9°C (chamber with leakage).  $\Delta T$  – boiling point difference resulting from air seepage into chamber.**



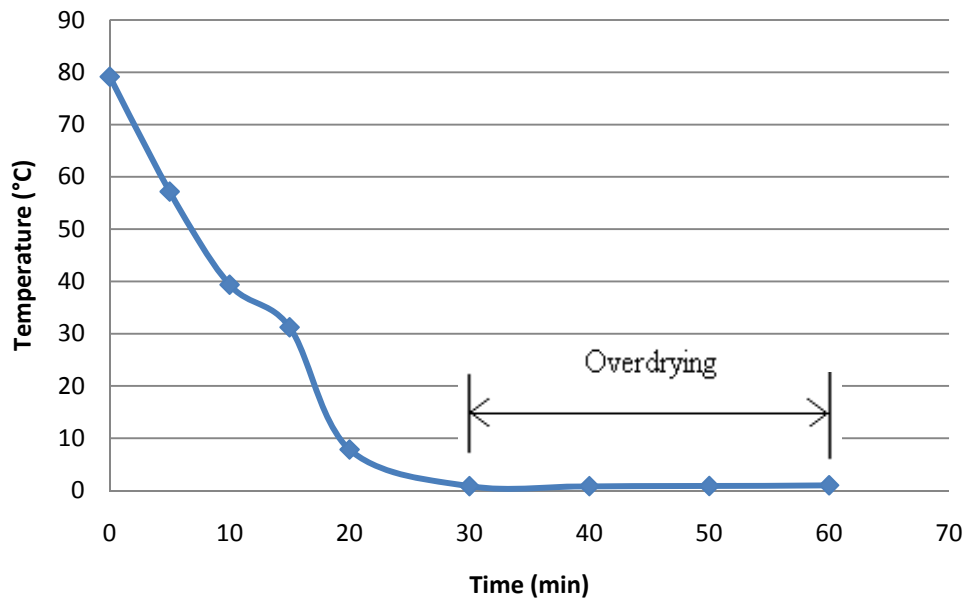
**Figure 10. Temperature change of water versus time for boiling point confirmation experiment under -25 kPa with an average evaporation temperature of 87.9°C (chamber with leakage).  $\Delta T$  – boiling point difference resulting from air seepage into the chamber.**

After the first series of experiments with the boiling point, several drying experiments were conducted with spent grain. It was observed that at a temperature slightly above the corresponding boiling point, spent grain samples were dried within 30 minutes in comparison to experiments by Thomkapanich et al. (2007) where the minimum drying time of 60 minutes was recorded. Figure 11 and 12 are the examples of my tests where samples were over-dried after 30 – 40 minutes. This again indicates possible air seepage into the system. Further experiments with different drying temperatures (Appendix A.3) confirmed that drying occurred at temperatures below the theoretical boiling points of water at the corresponding pressure. For example, at -20 kPa, drying occurred at an average temperature of 86.4°C (Figure 13), while at -25 kPa, drying occurred at an average temperature of 90.5°C (Figure 14). Both average temperatures

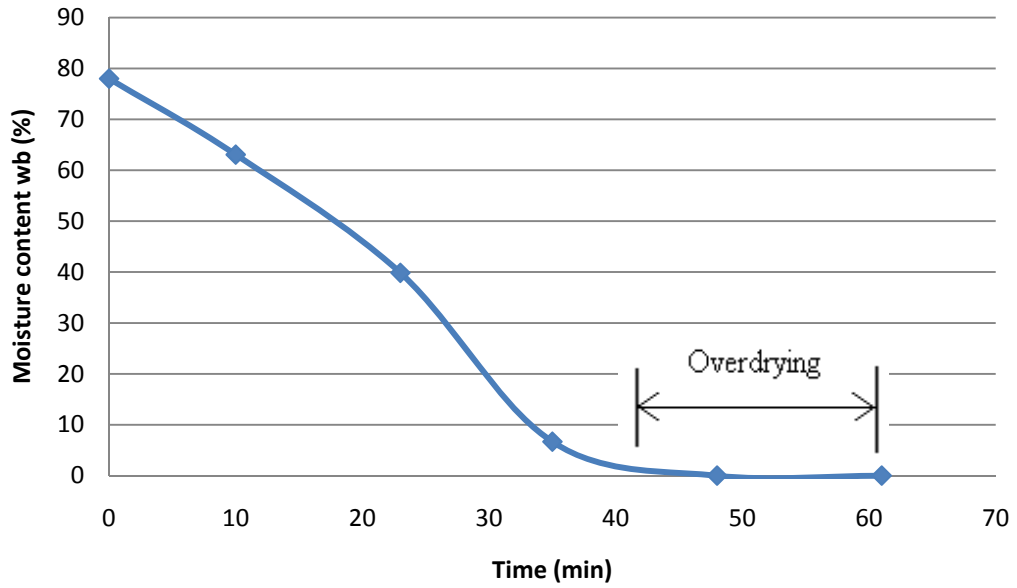
were below the theoretical boiling points (94 and 92 °C at the pressure of -20 and -25 kPa respectively).



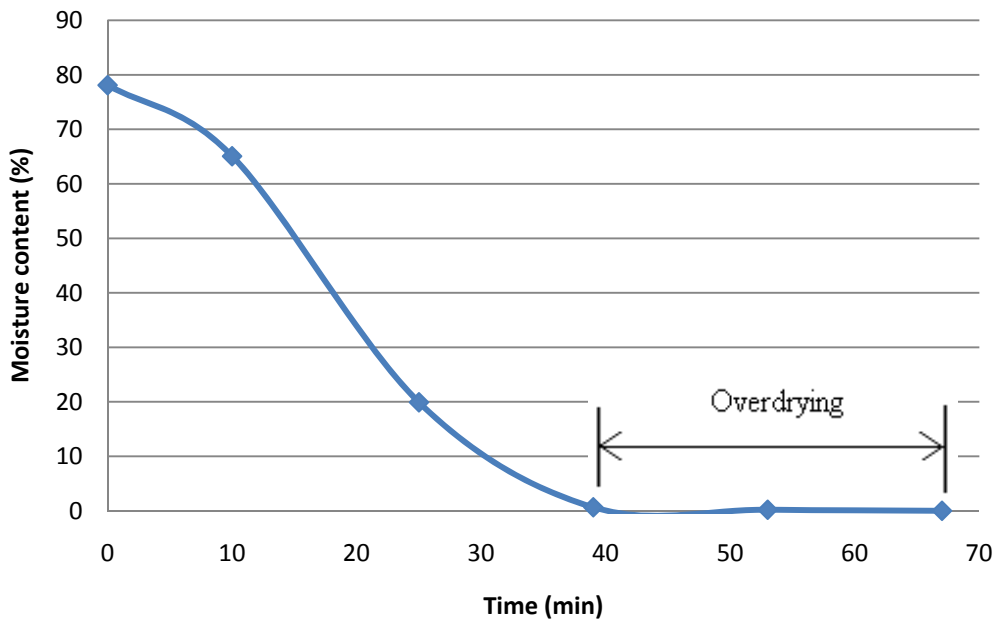
**Figure 11. Moisture content (wb) of sample versus time under vacuum pressure of -20 kPa with an overall average drying temperature and SS velocity of 94.8°C and of 0.178 m/s m/s respectively. Data obtained by the multiple-experiment method.**



**Figure 12. Moisture content (wb) of sample versus time under vacuum pressure of -25 kPa with an overall average drying temperature and SS velocity of 92.9°C and of 0.190 m/s respectively. Data obtained by the multiple-experiment method.**



**Figure 13. Moisture content (wb) of sample versus time under vacuum pressure of -20 kPa with an average drying temperature of 86.4°C and a SS velocity of 0.111 m/s. Data obtained by the single-experiment method.**



**Figure14. Moisture content (wb) of sample versus time under vacuum pressure of -25 kPa with an average drying temperature of 90.5°C and a SS velocity of 0.181 m/s. Data obtained by the single-experiment method.**

After some investigation, it was discovered that the chamber did not hold negative pressure when sealed. Following exposure to the vacuum, the valves (#10a and #10b, Figure 4) were closed and the vacuum pump was turned off to maintain negative pressure within the chamber. However, the pressure rose close to that of atmospheric. Therefore, a modification was made to the system, ensuring no vacuum would be lost due to the possibility of faulty valves. Two additional ball valves (#3e and #3f) were installed between the chamber and the 3-way bypass valves, one located before the inlet and the other following the outlet of the drying chamber. The additional valves (referred to as sealing valves) were used to completely seal the chamber, resulting in a closed volume.

After installation of the additional sealing valves, an experiment was conducted to determine if faulty valves were the cause of the lower experimental boiling points. To ensure that the chamber was completely sealed, the vacuum pump was turned on to reduce the pressure within the drying chamber, and then inlet and outlet valves as well as sealing valves were closed to maintain the vacuum without the pump. It was observed that the pressure within the chamber gradually rose to atmospheric pressure via the pressure gauges, indicating that the chamber was not completely sealed. As additional sealing valves, with the ability to prevent the leakage of water, oil and gas, were installed, the possibility of leakage due to faulty valves was excluded. Therefore, it was evident that the chamber contained at least one cavity, drawing air into the chamber.

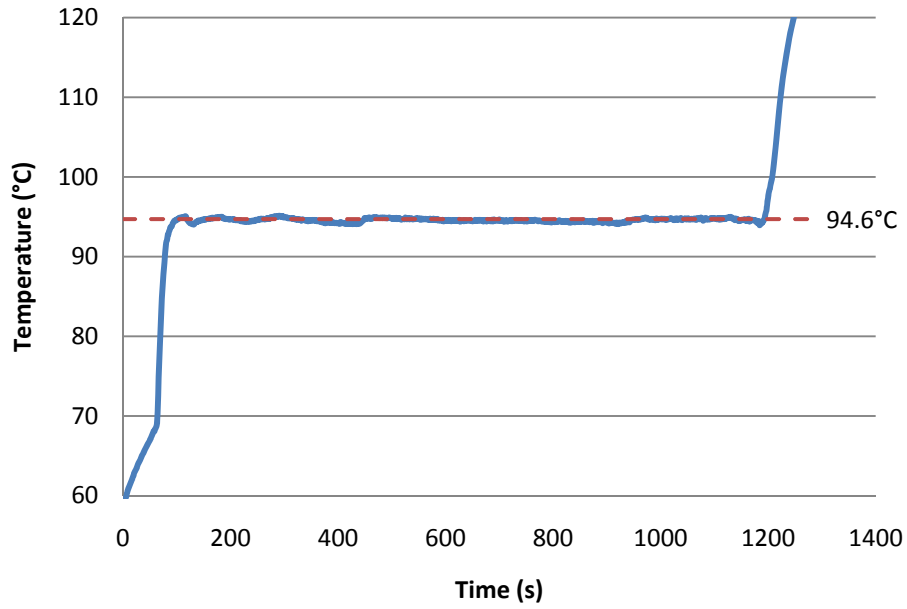
One method used to determine the location of the cavities in the chamber was placing droplets of water onto every joint and point of attachment on the chamber and the pipes between the sealing valves while the vacuum pump was turned on. Water droplets were drawn into the chamber where each cavity was located. Also, when the chamber

was allowed to reach a vacuum of -18 kPa or lower, the suction of air could be heard at points around the chamber, indicating a cavity. All cavities were sealed with a room temperature vulcanizing (RTV) silicone gasket maker that can stand temperatures up to 371 °C.

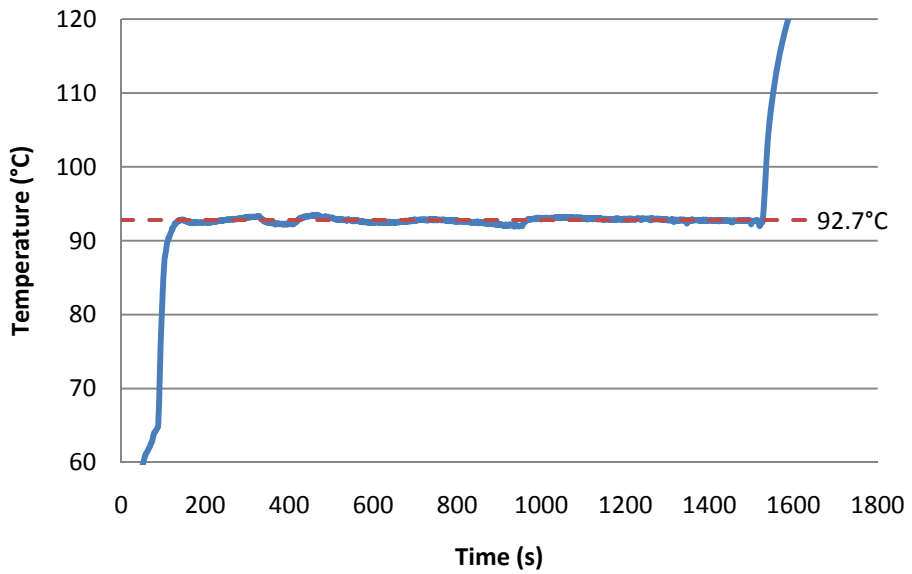
After all cavities were sealed and the chamber was able to maintain vacuum, an alternate boiling point experiment was conducted. As with the initial boiling point experiment, SS was allowed to flow through the drying chamber and the vacuum pump was turned on. The chamber pressure was adjusted accordingly. When the water (sample) reached a constant temperature (indicating the phase transition from water to steam) at the desired pressure, the two sealing valves were closed and the vacuum pump was turned off. Again, closing the sealing valves enabled the chamber to maintain the negative pressure without the vacuum pump or flow of steam.

With the heating tape as the only source of heat supply, the water temperature remained constant and was the same as that of theoretical boiling temperature at the corresponding pressure - average temperatures of 94.6 °C at -20 kPa (Figure 15 and Appendix A.4) and 92.7 °C at -25 kPa (Figure 16 and Appendix A.4). It was concluded that due to the unsealed chamber, air was drawn into the system, mixing with SS. As a result, the partial pressure was lowered due to presence of other gases. This caused a lower boiling temperature of water as compared to the theoretical boiling temperature of water.





**Figure 15. Temperature change of water versus time for boiling point confirmation experiment under -20 kPa (chamber without leakage).**

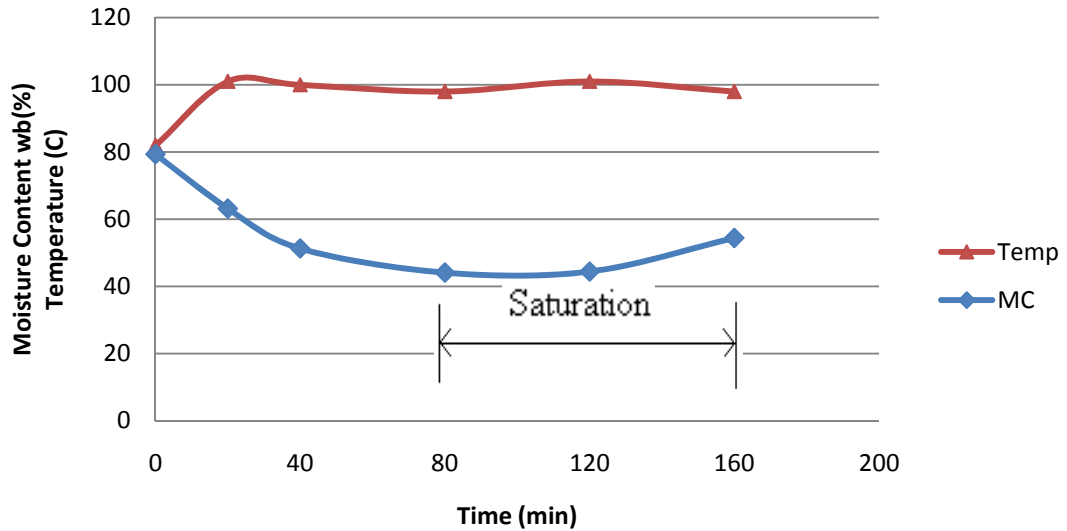


**Figure 16. Temperature change of water versus time for boiling point confirmation experiment under -25 kPa (chamber without leakage).**

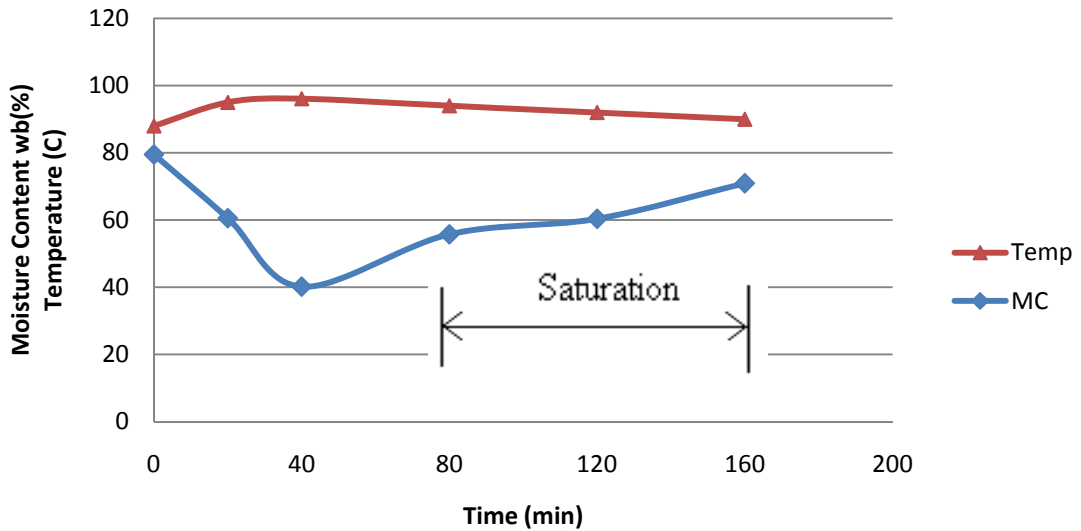
### 4.1.3 Sudden temperature drop

While conducting experiments to determine the falling rate and constant rate of drying, many experiments of different lengths were completed. Samples were dried for 20, 40, 80, 120 or 160 minutes at temperatures between 95 °C and 115 °C at -20kPa and at 95 °C and -25kPa, as well as 160 minutes at 100 °C and -20 kPa, weighing at 20, 40, 80, 120 and 160 minutes. The data were analyzed and graphed to determine the drying trends of the samples. It was observed that saturation of the samples often occurred during the longer experiments. In most cases, saturation would occur during the 80 to 160 minute experiments. Since both the boiler and vacuum pump maintained a constant pressure throughout all experiments, it was suspected that the superheater was the cause of the saturation.

When conducting experiments greater than 40 minutes, the occurrence of saturation was consistent. Repeatedly, a drastic temperature drop in both the sample and inlet was observed after 60 to 70 minutes of drying. The sample temperature would drop slightly below the boiling point for the corresponding pressure, indicating that condensation was occurring. Occasionally, the inlet temperature would drop as much as 30 °C, falling to the same temperature as the sample. This indicated rather than superheated steam, saturated steam was flowing to the chamber. This caused condensation to occur on the surface of the sample, increasing its moisture content (Figure 17, 18 and Appendix 5). The inlet temperature would remain at the reduced temperature for up to 10 minutes before rising again. As the boiler consistently generated steam and the vacuum pump supplied a constant pressure, the superheater was examined to obtain a possible cause for the drop in temperature.



**Figure 17. Moisture content (wb) of DSG versus time under vacuum pressure of -20 kPa - intended average drying temperature of 95°C and a SS velocity of 0.177 m/s (saturation occurred after 40 minutes of drying).**



**Figure 18. Moisture content (wb) of DSG versus time under vacuum pressure of -20 kPa - intended average drying temperature of 100°C and a SS velocity of 0.186 m/s (saturation occurred after 80 minutes of drying).**

Due to the high heat output of the superheater, the unit was turned down to the lower settings (79 - 121 °C) within the first few minutes of drying to maintain a fairly low sample temperature throughout the experiments. Since the desired sample temperature was between 95 and 115 °C for the experiments, the superheater remained at the lower settings for the majority of the drying time. While the flow of steam maintained a high temperature throughout most of the experiment (inlet temperature of about 120 °C), both the sample and inlet temperatures declined rapidly, resulting in saturation of the sample. The initial plunge in temperature was often observed after approximately 60 - 70 minutes of drying and occasionally, a second drop would occur closer to the end of the 160-minute experiment. The sample temperature consistently fell slightly below the boiling point (for example, 93 - 94 °C at -20 kPa) and stabilized within roughly 30 seconds. The inlet temperature dropped approximately 10 - 15 °C, sometimes more, matching that of the sample.

Since the temperature change in the sample and inlet occurred suddenly without any indication, the cause was likely from a sudden change in the system. As the boiler and vacuum pump maintained constant pressures throughout the duration of the experiments, as seen on the installed pressure gauges, the superheater was inspected. Besides the boiler and the heating tape, the superheater was the only other unit through which steam passed before flowing into the chamber, and was responsible for heating the steam above the boiling point at the given pressures. Heating tape was tested and confirmed that it was in good working condition. As the inlet temperature displayed superheated steam temperatures, the heating unit within the superheater was assumed to be in good working condition.

Given that the heating unit within the superheater appeared to be in working order, the orientation of the superheater was examined to find a possible cause for the drop in temperature. The superheater was oriented on its axial side, resulting in an upward flow at the inlet then horizontal through the heater and outlet. The orientation of the superheater was suspected to allow an accumulation of water within the unit since gravity would allow steam to settle at the bottom of the horizontal heating cavity. If water were to accumulate within the superheater, the water would eventually rise to a level that would inhibit the transition of saturated steam to superheated steam as superheated steam cannot exist in the presence of water. The flow of saturated steam through the drying chamber would cause a drop in both sample temperature and inlet temperature, adding condensation to the sample. The flow of steam would resume its superheated state once the accumulated water evaporated.

As a means to verify if the superheater was accumulating steam condensate, superheated steam was bypassed from the drying chamber during an experiment, then, the vacuum pump was turned off. The ball valve located at the inlet of the superheater was opened immediately, draining approximately 20 mL of water from the superheater. To further validate the accumulation of water as the cause of temperature drop, the superheater was drained immediately following an incident in which a drop in sample temperature and inlet temperature occurred. Unfortunately, the problem of sudden temperature drop did not disappear, but the time required to reach the desired temperature after the temperature drop was substantially smaller compared to that of the superheater without drainage. The temperature remained at the reduced temperature for

approximately 5 minutes before rising, while it took 10 minutes was taken when water was allowed to sit in the superheater.

After the accumulation of water within the superheater was confirmed, the unit was either drained after bypassing steam from the drying chamber or in conjunction with the intervals at which the sample was weighed to avoid any effect on the sample. The superheater was drained every 20 minutes of drying, released as much as 112 mL of water, reducing the occurrence of saturation of the sample.

Further investigation led to the discovery of air seepage caused by the vacuum breaker. When the vacuum pump was turned on, the desired condition was to have only steam generated by the boiler to be drawn into and pass through the chamber. It was found that a small amount of air was drawn into the system through the vacuum breaker irregularly. This irregular air seepage caused unsteady flow of steam, which lowered the velocity of steam and choked the superheater. Due to the unstable amount of steam passing through the piping, the chamber temperature varied. When enough superheated steam was flowing through the chamber, its temperature was stable. When the superheater was choking, not enough steam was going through. After a certain point (60 - 70 minutes of drying), a large amount of steam was accumulated where before the superheater, and it rushed through the superheater, passed through the chamber without being superheated. Therefore, steam condensed within the chamber. Thus, a sudden temperature drop of the chamber was observed.

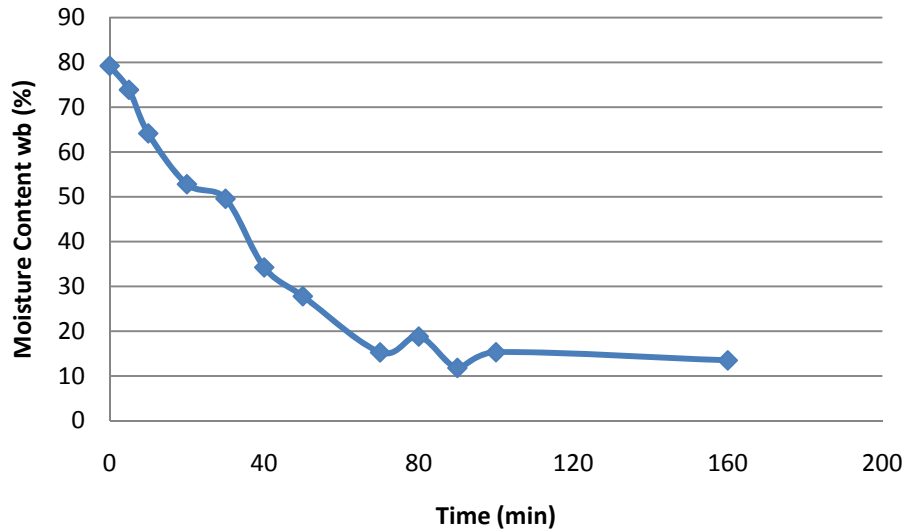
Further experiments were conducted with the valve connecting the vacuum breaker to the piping closed. No temperature drop was observed. The valve was only opened when the experiment was finished to allow for the end vacuum to be dissipated.

#### **4.2 Drying experiments under vacuum**

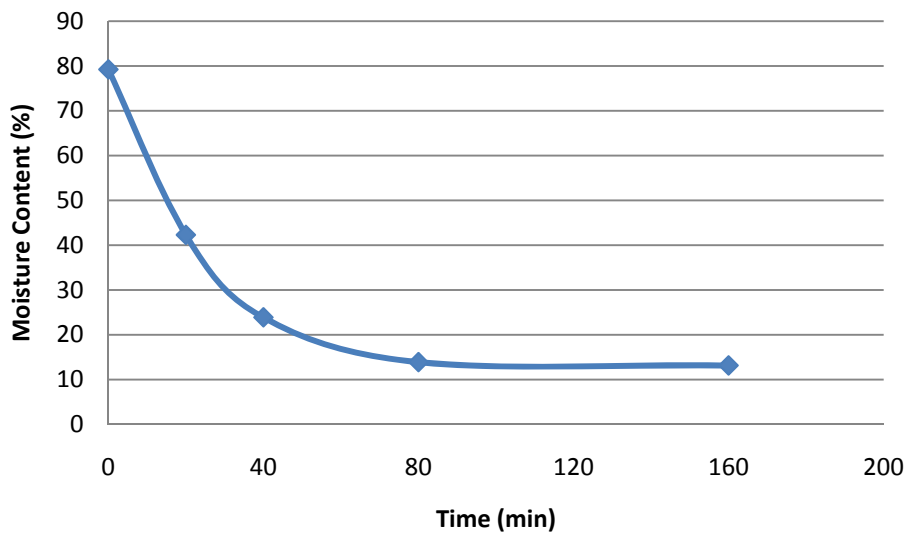
After confirming the system worked as expected under the desired conditions, experiments were conducted with spent grain. The multiple-experiment and single-experiment methods were used.

The multiple-experiment method was used for experiments conducted during the earlier stage. Each point on the sample moisture content versus drying time curve represented one experiment for the corresponding drying time (except for the ones at time equal to 0 minute). For example, a point on the 120 minutes drying time line meant that the sample was processed for a complete 120 minutes without interruption, and the next drying interval was 160 minutes, a new sample was used and being processed for another 160 minutes. Figure 19 and 20 were composed of 11 and 5 separate experiments respectively. This method of constructing a curve was extremely time consuming. For example, the total time to construct Figure 19 was the sum of all time intervals, which was equal to 655 minutes – almost 11 hours, while the total time to construct Figure 20 was equal to 300 minutes – 5 hours.

Due to high time consumption for experiments, the single-experiment method was used for all the remaining experiments. All experiments using this method had a maximum drying time of 160 minutes. Figure 21 and 22 were graphs constructed with this method.

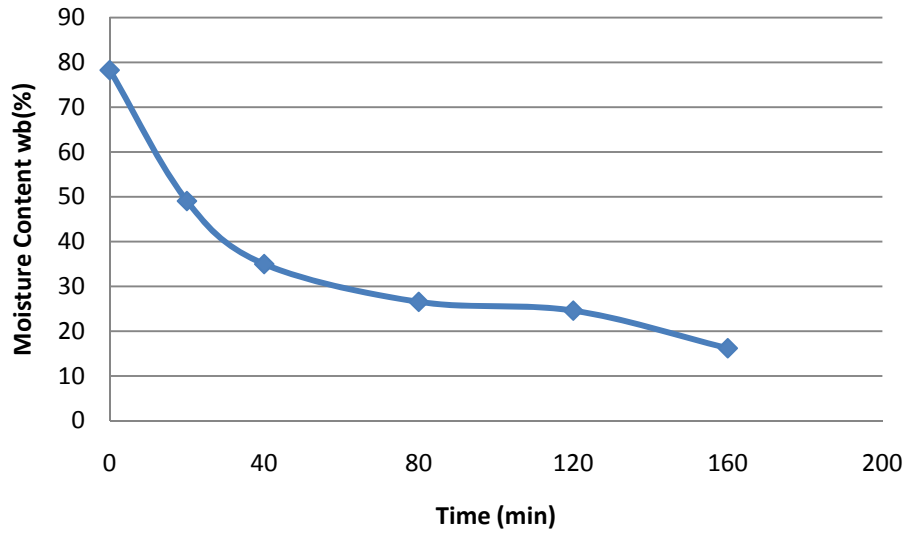


**Figure 19. Moisture content (wb) of DSG versus time under vacuum pressure of -20 kPa with an overall average drying temperature of 95.4°C and a SS velocity of 0.204 m/s. Data obtained by the multiple-experiment method.**

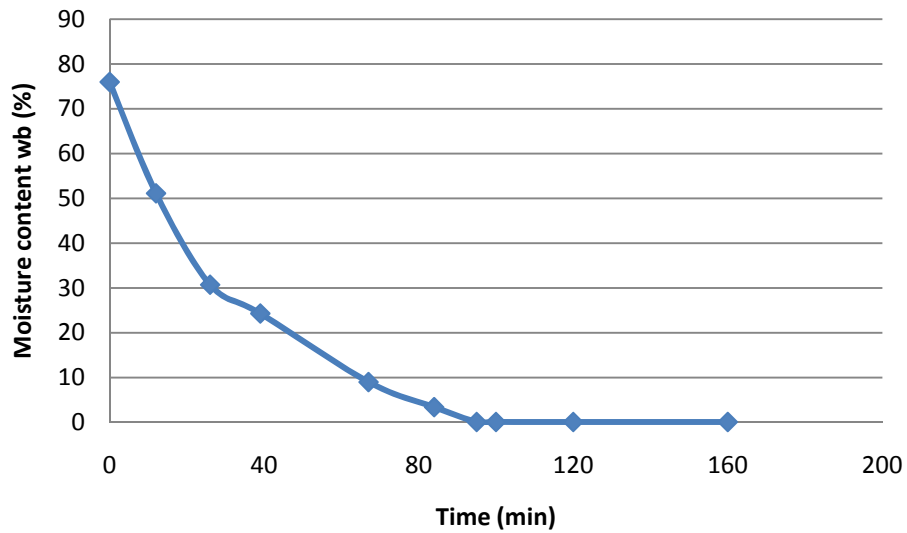


**Figure 20. Moisture content (wb) of DSG versus time under vacuum pressure of -20 kPa with an overall average drying temperature of 105.6°C and a SS velocity of 0.183 m/s. Data obtained by the multiple-experiment method.**





**Figure 21. Moisture content (wb) of DSG versus time under vacuum pressure of -20 kPa with an overall average drying temperature of 101.5°C and a SS velocity of 0.193 m/s. Data obtained by the single-experiment method.**



**Figure 22. Moisture content (wb) of DSG versus time under vacuum pressure of -20 kPa with an overall average drying temperature of 114.6°C and a SS velocity of 0.213 m/s. Data obtained by the single-experiment method.**

It was observed that with higher drying temperature, the sample dried faster. This was further confirmed by conducting experiments with higher drying temperatures (Appendix A.6). The maximum SS velocity flowing through the chamber was 0.289 m/s. Experiments with a higher velocity were attempted, but the superheater was not able to superheat all the steam passing through it and condensation was observed. The minimum SS velocity of 0.100 m/s was obtained as the steam started to pass through the steam valve when it was opened by turning it by 25 °.

## 5 CONCLUSIONS

The newly designed low pressure superheated steam processing system was fabricated at the University of Manitoba. Testing of the system was carried out and three major design issues were identified. These issues were resolved after several modifications were made to the first design. The problem of excessive suction of vacuum pump was resolved by installation of a globe valve at downstream of the inlet piping, giving control of level of suction. The lower-than-theoretical boiling point of water led to discovery of unsealed chamber. Sealing of all cavities using a RTV silicone gasket maker prevented air from entering the system, and allowed SS vacuum drying in chamber. Thus drying occurred at temperatures equal to the theoretical boiling points of water at the corresponding pressure. The sudden temperature drop showed evidence that saturation occurred during the drying process. Draining the superheater regularly during the experiment greatly reduced the amount of time needed for sample and inlet temperatures to rise to the desired temperature above the boiling point. Closing the valve connecting the vacuum breaker to the piping prevented air seepage and chocking of superheater, which maintained a steady flow of steam. Therefore, the problem of sudden temperature drop was eliminated.

After modifications to the system, experiments were conducted and DSG was successfully dried under different vacuum pressures of -25 and -20 kPa and temperatures between 95 to 115 °C, with a maximum drying time of 160 minutes. The SS velocity passing through the drying chamber ranged from 0.100 to 0.289 m/s.

## **6 RECOMMENDATIONS FOR FUTURE RESEARCH**

### **6.1 Design improvements**

As the system fabricated in this project was in pilot scale, the volume of drying chamber was only enough to hold one sample container. A larger drying chamber that holds a bigger sample container or multiple sample containers can be used to obtain a more accurate result with larger sample size. Drying multiple samples in one experiment gives higher system efficiency in terms of time.

During the stage of assembling the system, inserting pipe connectors in between components is one of the essential parts needed for the system when putting components together. When a component of the built system is not working properly or broken down, it needs to be taken out and repaired or replaced. With pipe connectors, simply loosening the ones that are nearest to the component that needs to be taken out, and then the component is no longer linked to the system and can be easily disconnected. Connecting the component back to the system can be done simply by tightening the pipe connectors. Without pipe connectors close to a defective component, all the piping and other components located before the next connector have to be taken out to disconnect the defective component, which requires substantial amount of time and labor.

Some components of the system can be computerized. As the steam-flow and air valves were monitored and controlled manually, an operator was needed to be near the system during the entire experimental period. This made the two valves potential components that could be computerized. Automated control and monitoring of these

valves eliminate the need of manual monitoring and control, which greatly reduces the time needed for conducting an experiment.

## **6.2 Suggestion for future research**

As only a very limited research has been done regarding low pressure SS drying, some of the related research areas can be studied more in depth such as the design process and fabrication of a low pressure SS drying system. Description and purpose of components used in fabricating the system can be included. Additional research can also be done to examine various parameters in food processing. This may include the quality of dried product (including chemical, microbial, and nutritional changes for food product), range of inversion temperature, range of velocity flow for optimum drying condition, design of the operating system, energy consumption levels, and a variety of experimental materials used such as meat or other fruits and vegetables in the food processing area.

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## APPENDICES

### Appendix A.1 Moisture content (mc) determination

$$mc = \frac{m_{H_2O}}{m_t} \times 100\% \quad \text{moisture content (wb)}$$

Where  $m_d$  = dry mass of the product

$m_{H_2O}$  = mass of water in the product

$m_t$  = total mass of product:  $m_t = m_{H_2O} + m_d$

## Appendix A.2

**Table A2-1** Experimental results for initial moisture content determination (before drying).

Drying temperature: 135 °C  
Total drying time: 2.5 h

Replicate	Mass (g)		
	Sample Container	Wet Sample	Dish + Sample(2.5h)
1	0.96	3.01	1.62
2	0.96	2.99	1.59
3	0.95	3.05	1.62
4	0.95	3.05	1.70
5	0.95	3.04	1.66
6	0.96	2.97	1.63
7	0.96	3.00	1.64
8	0.97	3.05	1.65
9	0.96	3.06	1.65
10	0.96	3.05	1.61
11	0.97	3.07	1.65
12	0.97	3.03	1.63

**Table A2-2 Experimental results for initial moisture content determination (after 2.5 hours of drying.)**

Replicate	m <sub>H2O</sub> (g)	m <sub>dry</sub> (g)	mc(wb) %	mc(db) dcml	Δmc(wb)	Δmc(wb) <sup>2</sup>
1	2.35	0.66	78.07	3.56	0.43	0.19
2	2.36	0.63	78.93	3.74	1.29	1.65
3	2.38	0.67	78.03	3.55	0.39	0.15
4	2.30	0.75	75.41	3.06	-2.23	5.00
5	2.33	0.71	76.64	3.28	-1.00	1.01
6	2.30	0.67	77.44	3.43	-0.20	0.04
7	2.32	0.68	77.33	3.41	-0.31	0.10
8	2.37	0.68	77.70	3.48	0.06	0.00
9	2.37	0.69	77.45	3.43	-0.19	0.04
10	2.40	0.65	78.68	3.69	1.04	1.08
11	2.39	0.68	77.85	3.51	0.21	0.04
12	2.37	0.66	78.21	3.59	0.57	0.33
Total:			931.77	Sum:		9.63
Average:			77.64			

mc (wb) = moisture content in wet basis

Δmc(wb) = deviation of each number from mean of sample

Sample Variance  $s^2 = \sum \Delta mc / (n-1) = 9.63/11 = 0.88$

Moisture content: 77.64 ± .88 %

### Appendix A.3

Experiments with different drying temperatures for confirmation of drying occurrence below boiling point with the corresponding pressure.

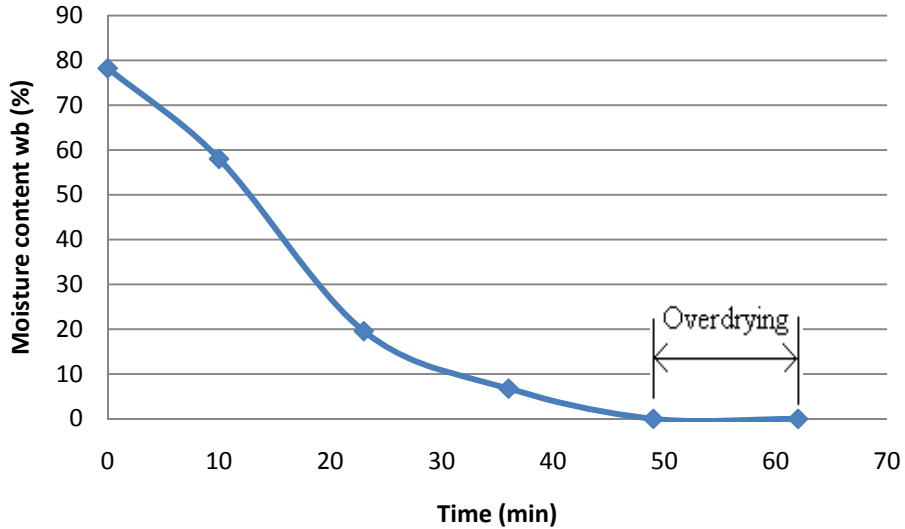


Figure A3-1. Moisture content (wb) of DSG versus time under vacuum pressure of -25 kPa with an average drying temperature of 92.9°C and a SS velocity of 0.139 m/s.

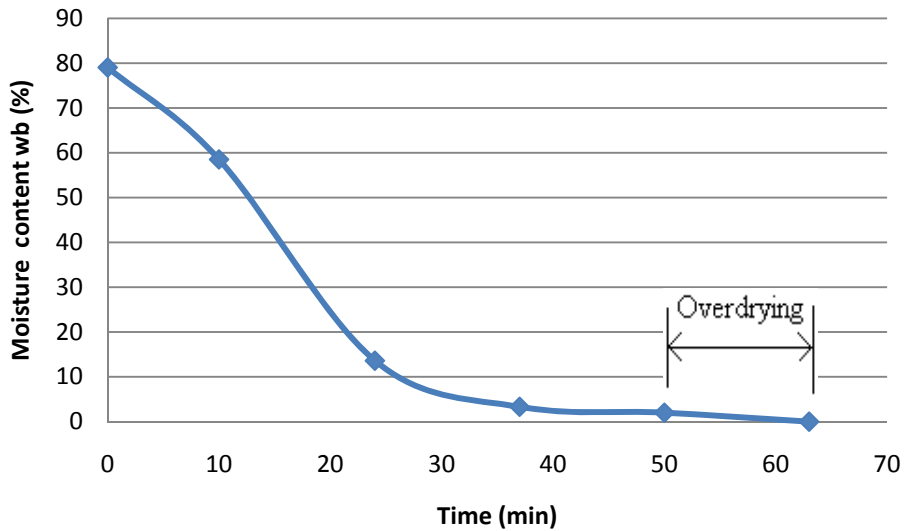
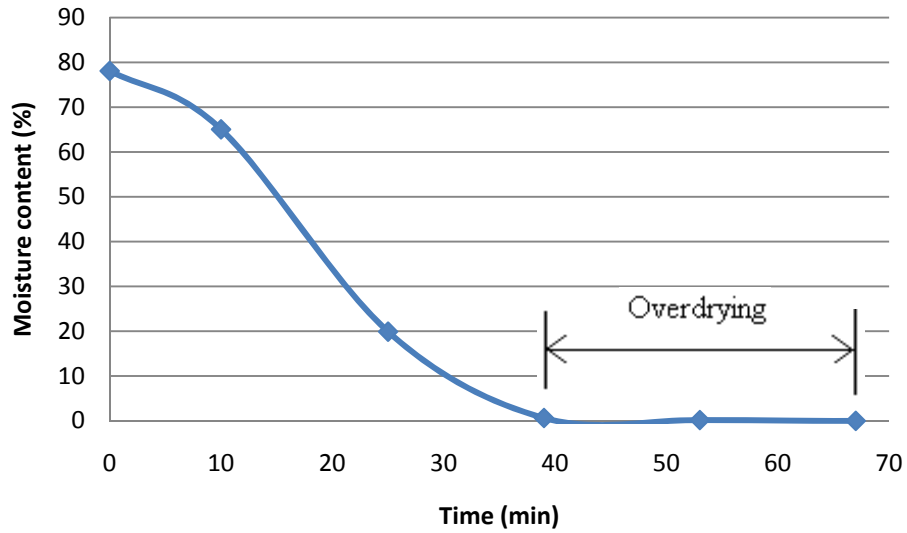
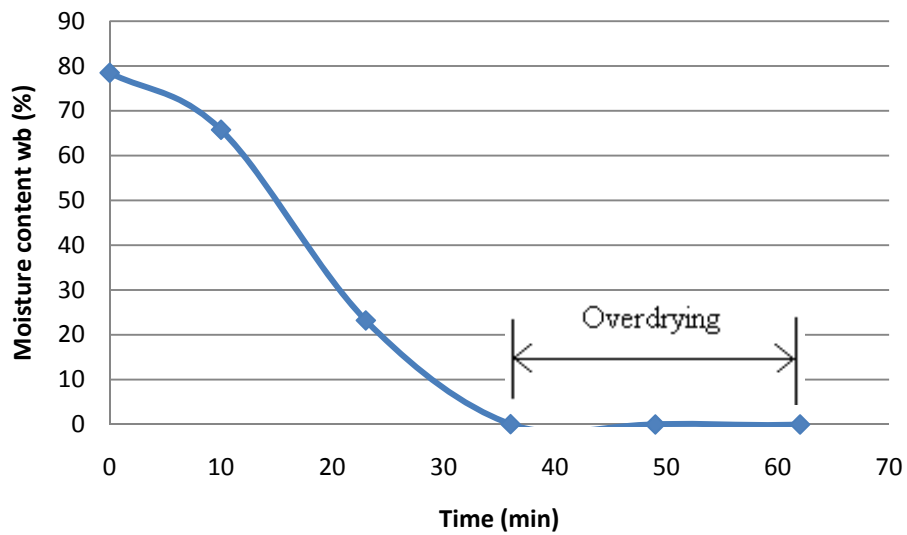


Figure A3-2. Moisture content (wb) of DSG versus time under vacuum pressure of -25 kPa with an average drying temperature of 92.7°C and a SS velocity of 0.112 m/s.

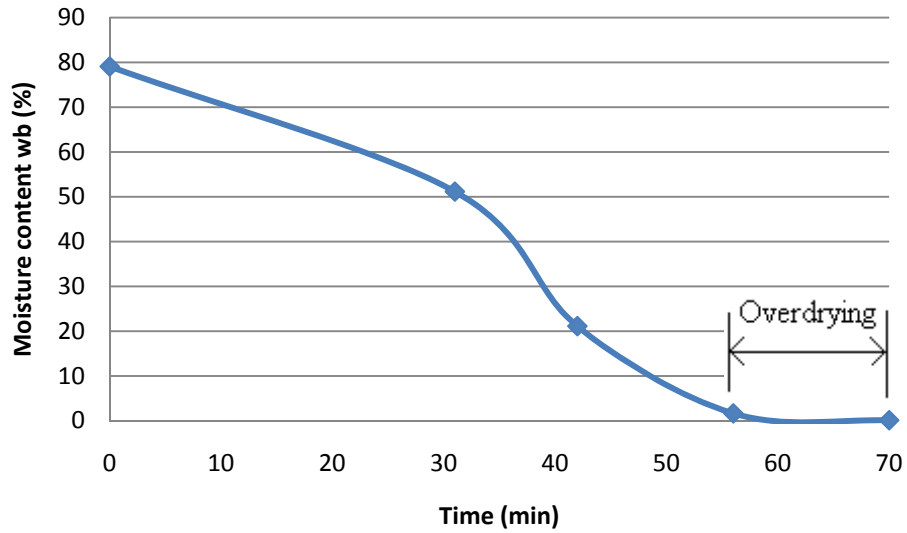


**Figure A3-3. Moisture content (wb) of DSG versus time under vacuum pressure of -25 kPa with an average drying temperature of 90.5°C and a SS velocity of 0.181 m/s.**

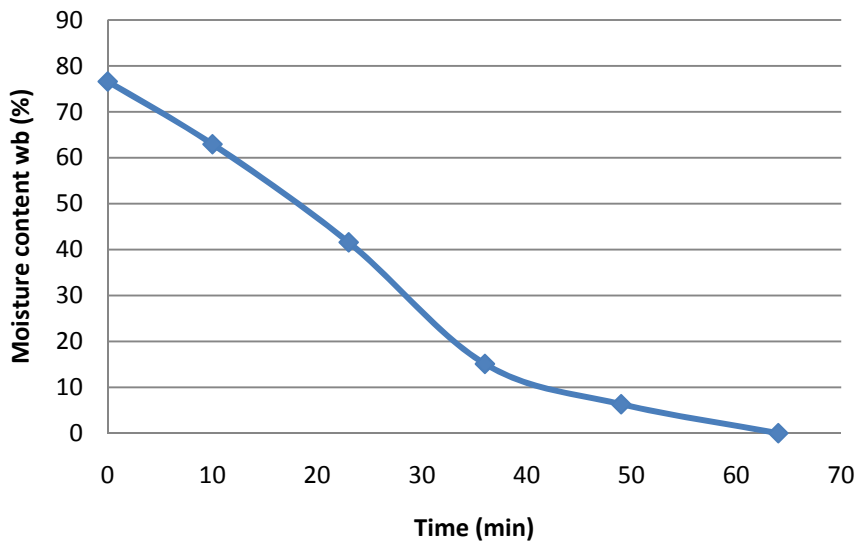


**Figure A3-4. Moisture content (wb) of DSG versus time under vacuum pressure of -25 kPa with an average drying temperature of 90.3°C and a SS velocity of 0.164 m/s.**

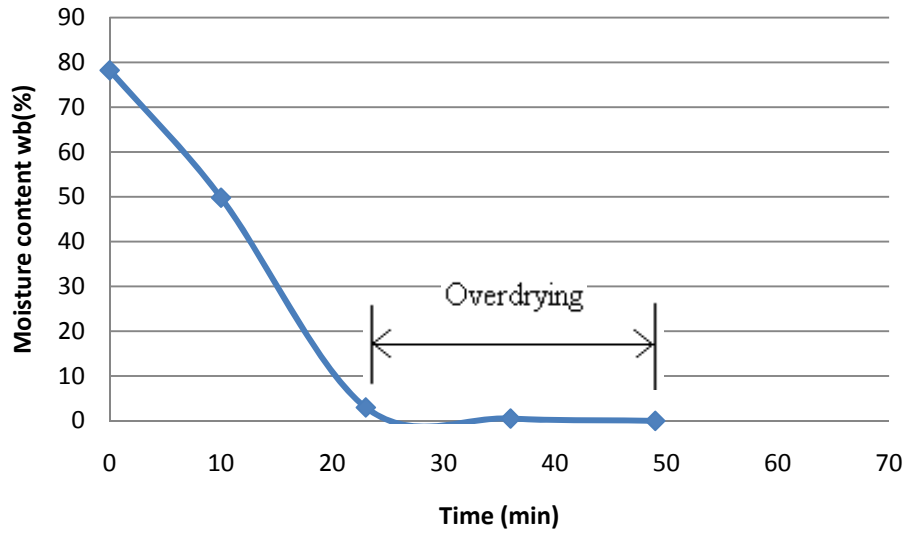




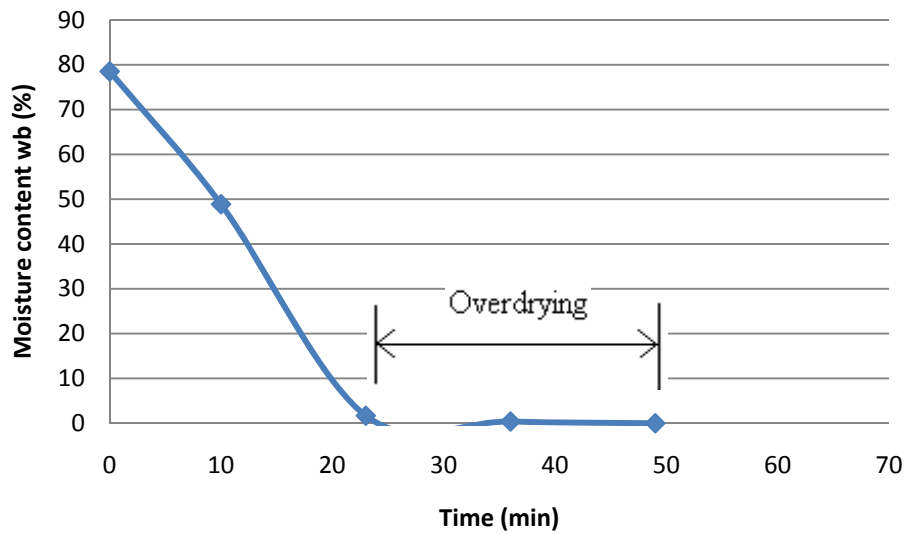
**Figure A3-5. Moisture content (wb) of DSG versus time under vacuum pressure of -20 kPa with an average drying temperature of 98.9°C and a SS velocity of 0.133 m/s.**



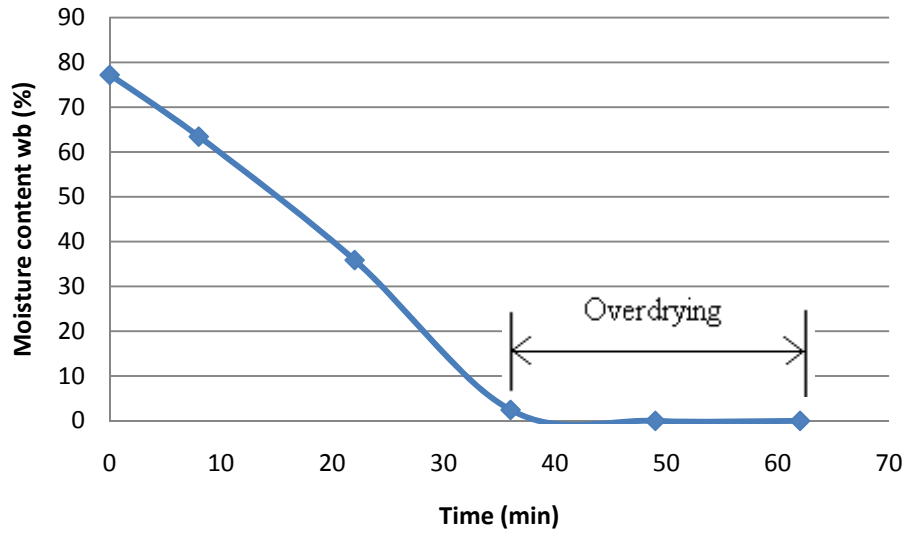
**Figure A3-6. Moisture content (wb) of DSG versus time under vacuum pressure of -20 kPa with an average drying temperature of 95.6°C and a SS velocity of 0.247 m/s.**



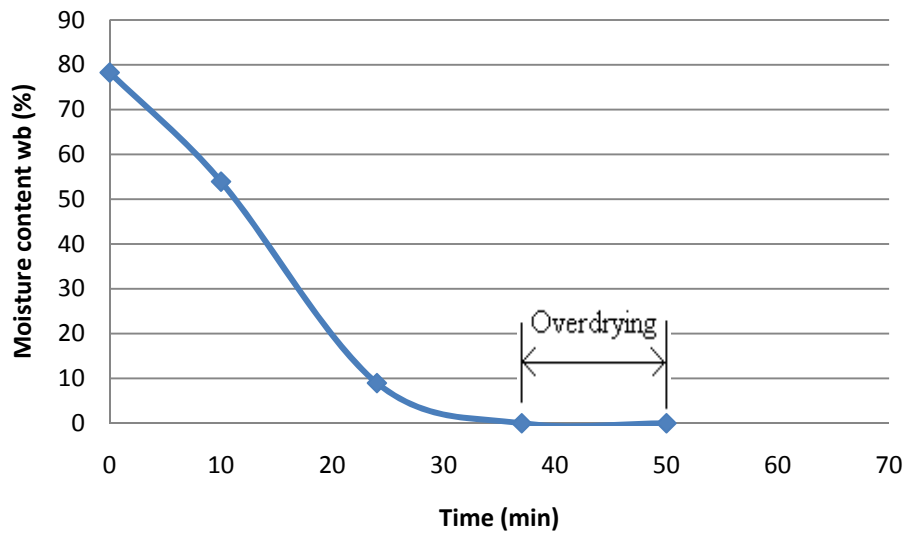
**Figure A3-7. Moisture content (wb) of DSG versus time under vacuum pressure of -20 kPa with an average drying temperature of 95.5°C and a SS velocity of 0.178 m/s. and a SS velocity of 0.247 m/s.**



**Figure A3-8. Moisture content (wb) of DSG versus time under vacuum pressure of -20 kPa with an average drying temperature of 95.0°C and a SS velocity of 0.232 m/s.**



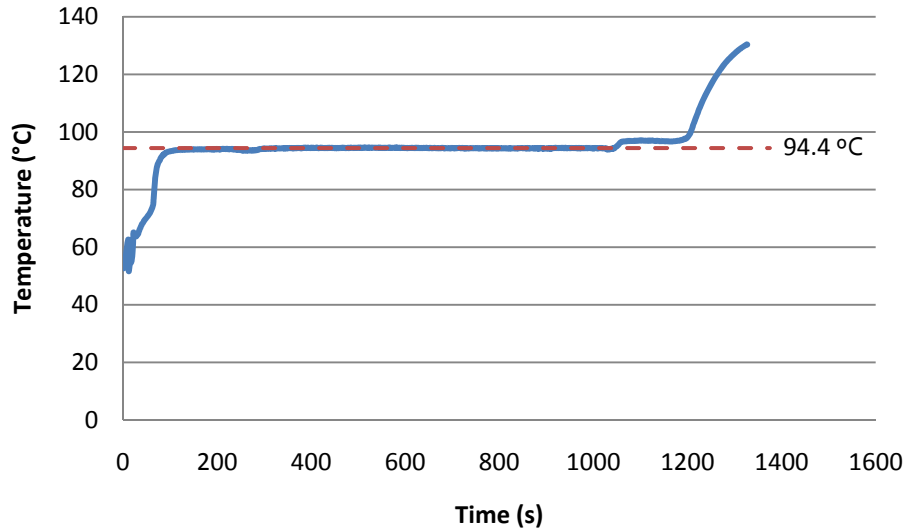
**Figure A3-9. Moisture content (wb) of DSG versus time under vacuum pressure of -20 kPa with an average drying temperature of 94.9°C and a SS velocity of 0.111 m/s.**



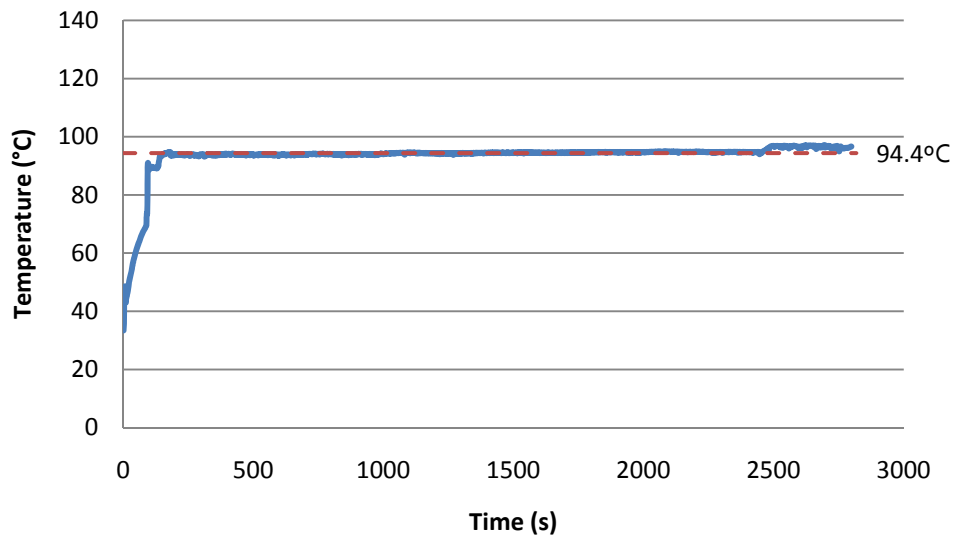
**Figure A3-10. Moisture content (wb) of DSG versus time under vacuum pressure of -20 kPa with an average drying temperature of 93.4°C and SS velocity of 0.186 m/s.**

## Appendix A.4

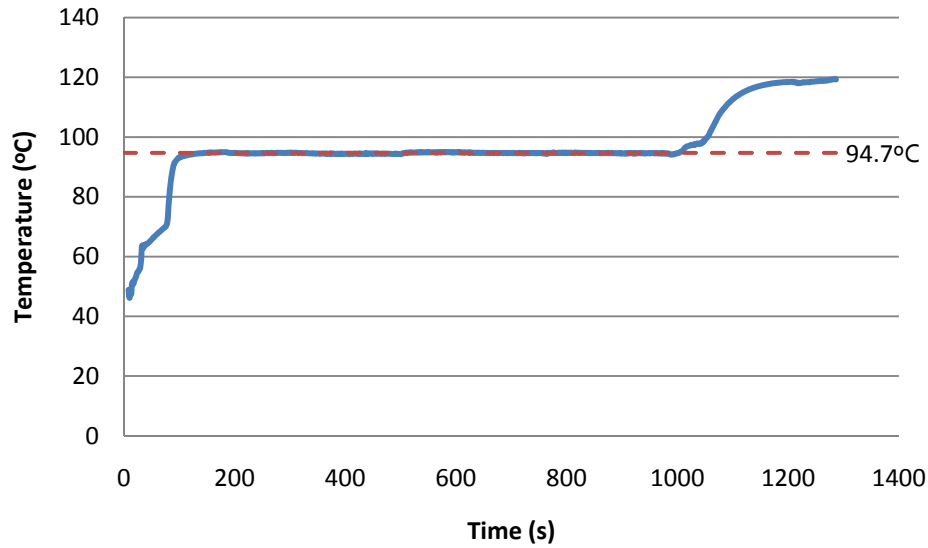
### Experimental results for boiling point determination.



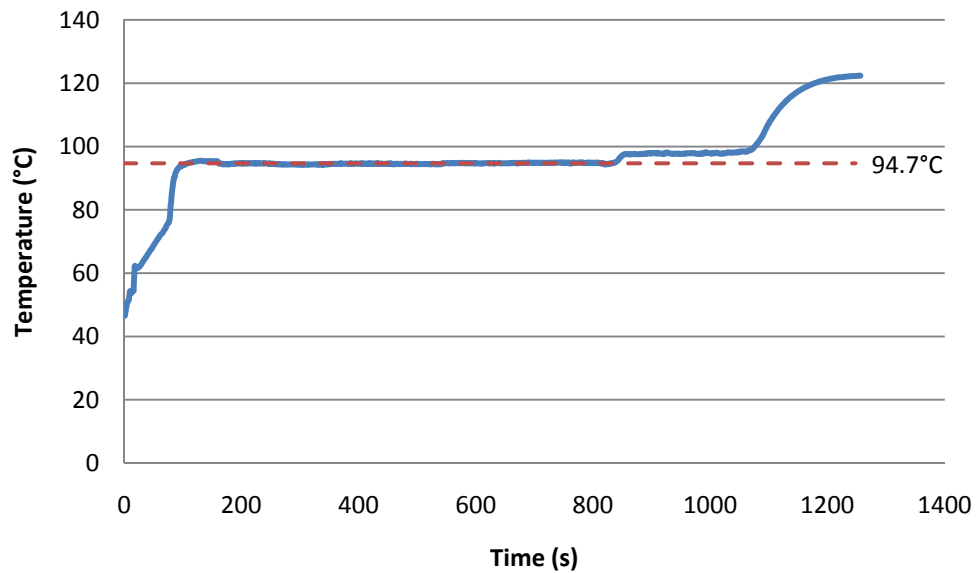
**Figure A4-1. Temperature change of water versus time for boiling point confirmation experiment under -20 kPa with an average evaporation temperature of 94.4°C (chamber without leakage).**



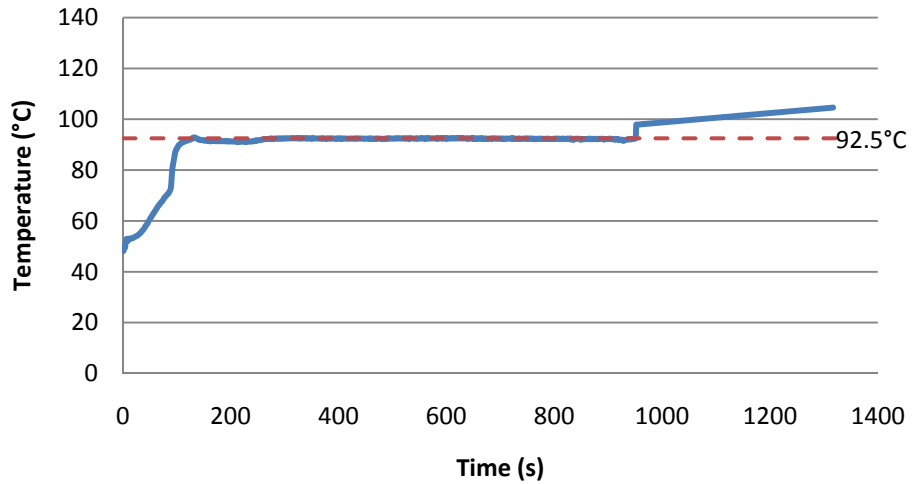
**Figure A4-2 Temperature change of water versus time for boiling point confirmation experiment under -20 kPa with an average evaporation temperature of 94.4°C (chamber without leakage).**



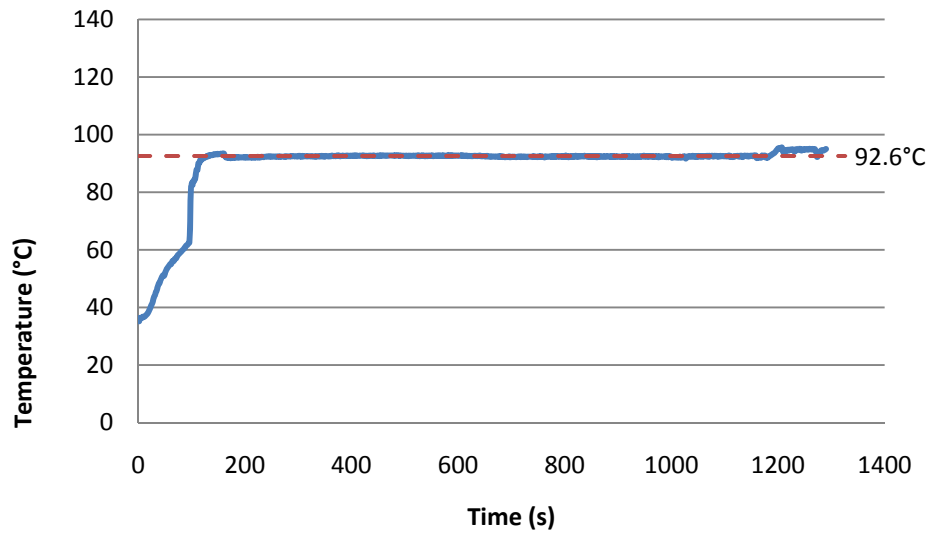
**Figure A4-3 Temperature change of water versus time for boiling point confirmation experiment under -20 kPa with an average evaporation temperature of 94.7°C (chamber without leakage).**



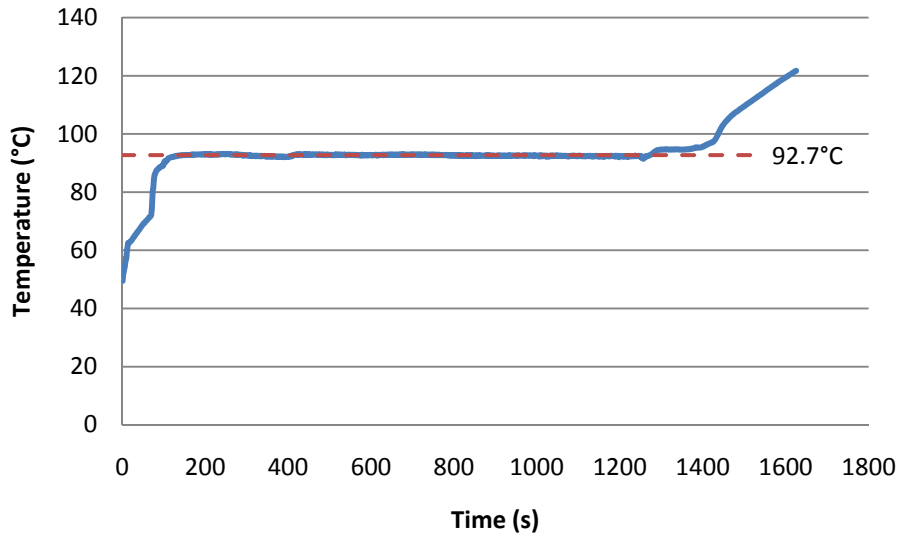
**Figure A4-4. Temperature change of water versus time for boiling point confirmation experiment under -20 kPa with an average evaporation temperature of 94.7°C (chamber without leakage).**



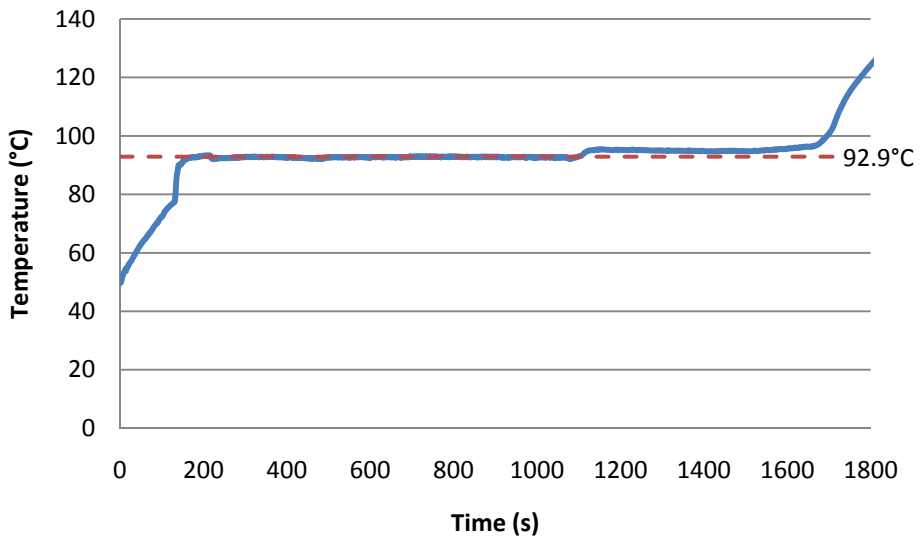
**Figure A4-5. Temperature change of water versus time for boiling point confirmation experiment under -25 kPa with an average evaporation temperature of 92.5°C (chamber without leakage).**



**Figure A4-6. Temperature change of water versus time for boiling point confirmation experiment under -25 kPa with an average evaporation temperature of 92.6°C (chamber without leakage).**

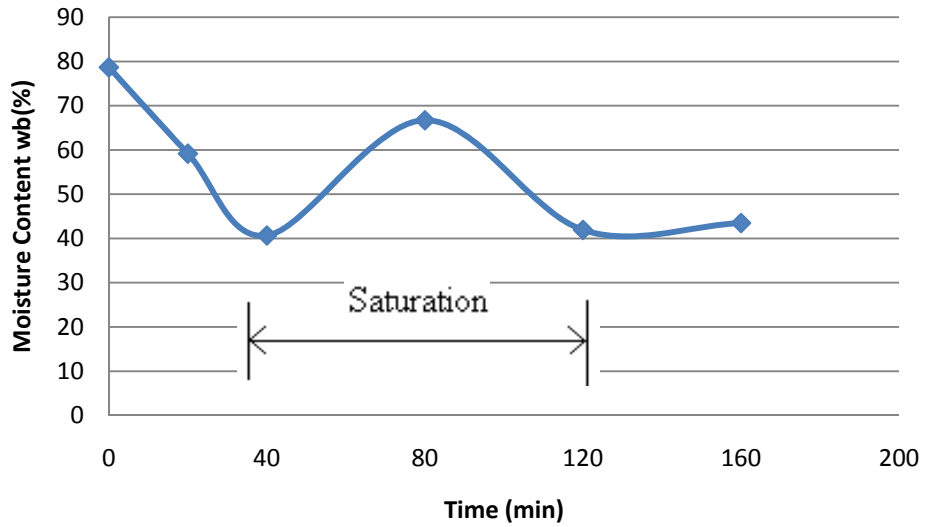


**Figure A4-7. Temperature change of water versus time for boiling point confirmation experiment under -25 kPa with an average evaporation temperature of 92.7°C (chamber without leakage).**

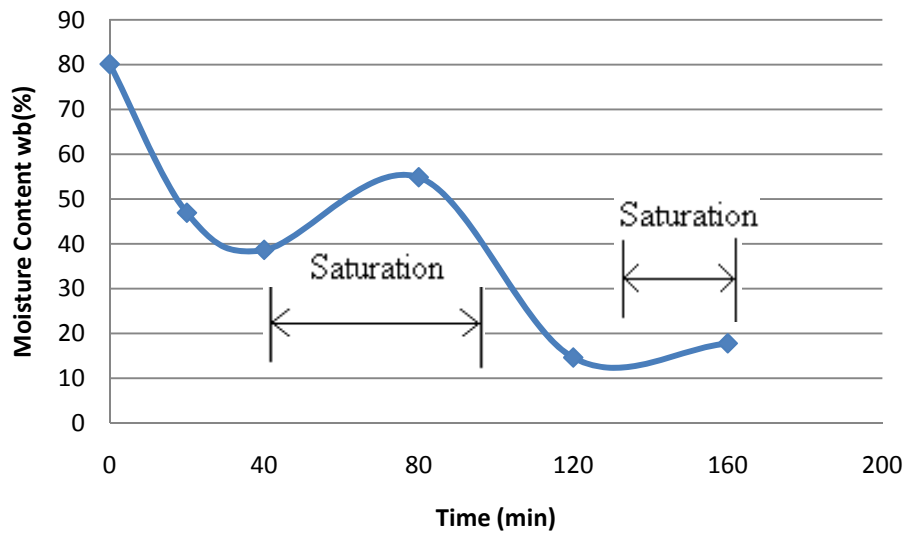


**Figure A4-8. Temperature change of water versus time for boiling point confirmation experiment under -25 kPa with an average evaporation temperature of 92.9°C (chamber without leakage).**

**Appendix A.5 Experimental results identifying condensation during drying process**

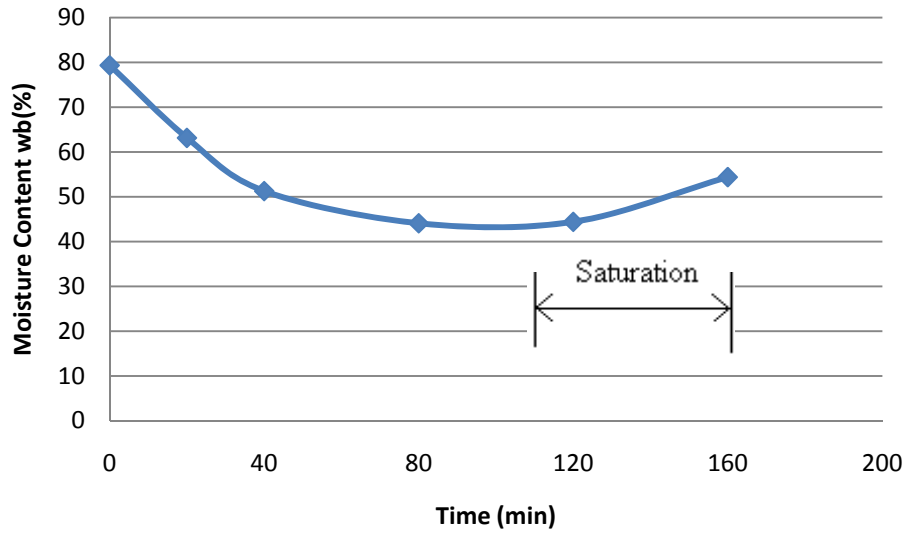


**Figure A5-1. Moisture content (wb) of DSG versus time under vacuum pressure of -20 kPa - intended average drying temperature of 95°C and a SS velocity of 0.167 m/s (saturation occurred after 40 minutes of drying).**

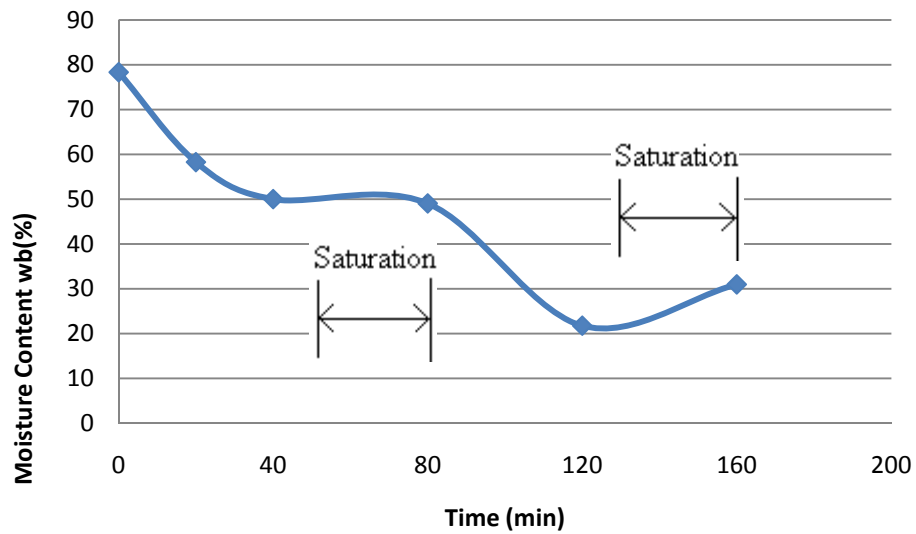


**Figure A5-2. Moisture content (wb) of DSG versus time under vacuum pressure of -20 kPa - intended average drying temperature of 100°C and a SS velocity of 0.192 m/s (saturation occurred after 40 minutes of drying).**

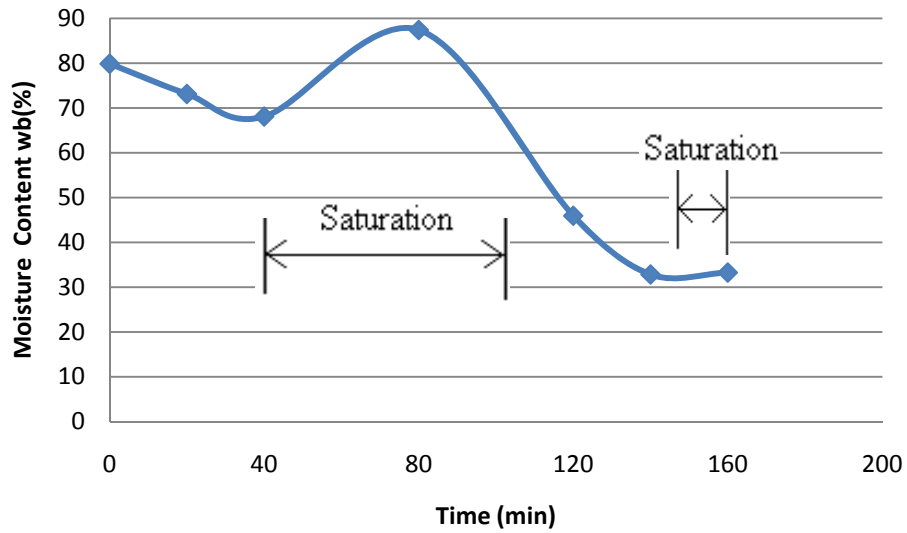




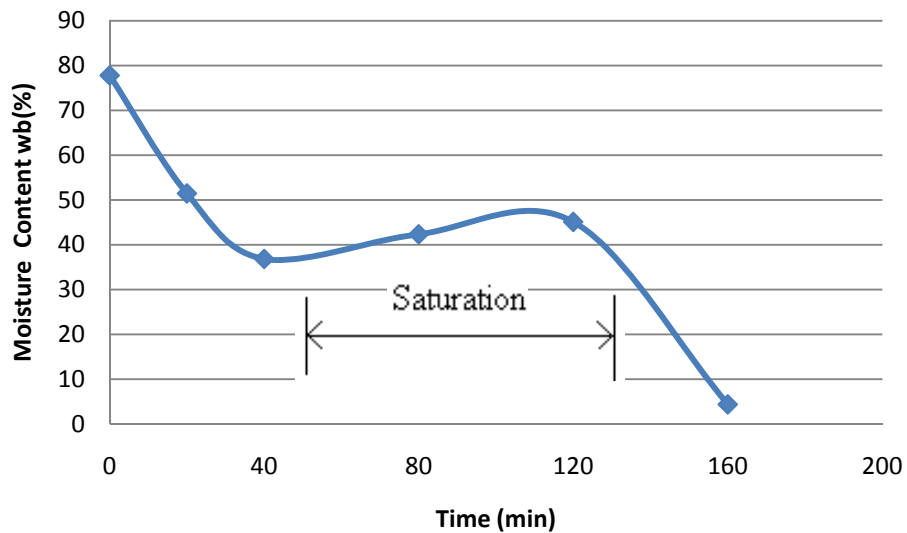
**Figure A5-3. Moisture content (wb) of DSG versus time under vacuum pressure of -20 kPa - intended average drying temperature of 100°C and a SS velocity of 0.159 m/s (saturation occurred after 110 minutes of drying).**



**Figure A5-4. Moisture content (wb) of DSG versus time under vacuum pressure of -20 kPa - intended average drying temperature of 100°C and a SS velocity of 0.175 m/s (Saturation occurred after 50 minutes of drying).**



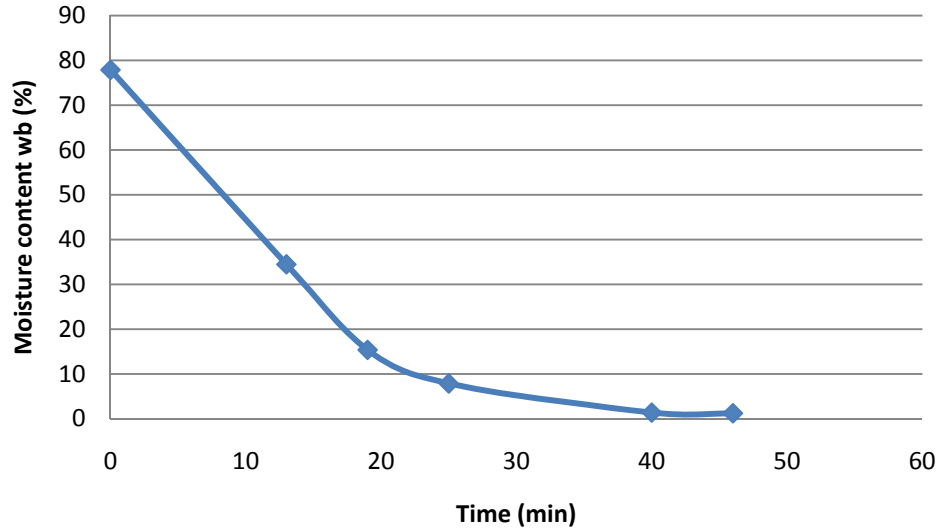
**Figure A5-5. Moisture content (wb) of DSG versus time under vacuum pressure of -20 kPa - intended average drying temperature of 100°C and a SS velocity of 0.177 m/s (Saturation occurred after 40 minutes of drying).**



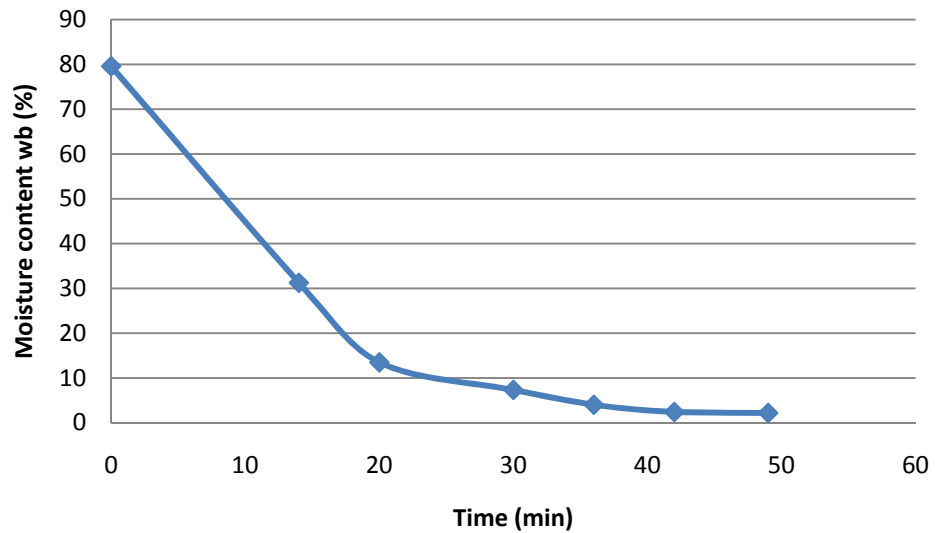
**Figure A5-6. Moisture content (wb) of DSG versus time under vacuum pressure of -20 kPa - intended average drying temperature of 105°C and a SS velocity of 0.188 m/s (Saturation occurred after 50 minutes of drying).**

## Appendix A.6

Experiments with different drying temperatures for observation of relationship between drying temperature and drying time.



**Figure A6-1. Moisture content (wb) of DSG versus time under vacuum pressure of -20 kPa with an average drying temperature of 155.9°C and SS velocity of 0.105 m/s.**



**Figure A6-2. Moisture content (wb) of DSG versus time under vacuum pressure of -20 kPa with an average drying temperature of 158.2°C and SS velocity of 0.100 m/s**

Appendix A.7

**OPERATION MANUAL FOR LOW PRESSURE SUPERHEATED STEAM SYSTEM**

This operation manual refers to Figure A.8. All components mentioned are in capital letters with the corresponding number.

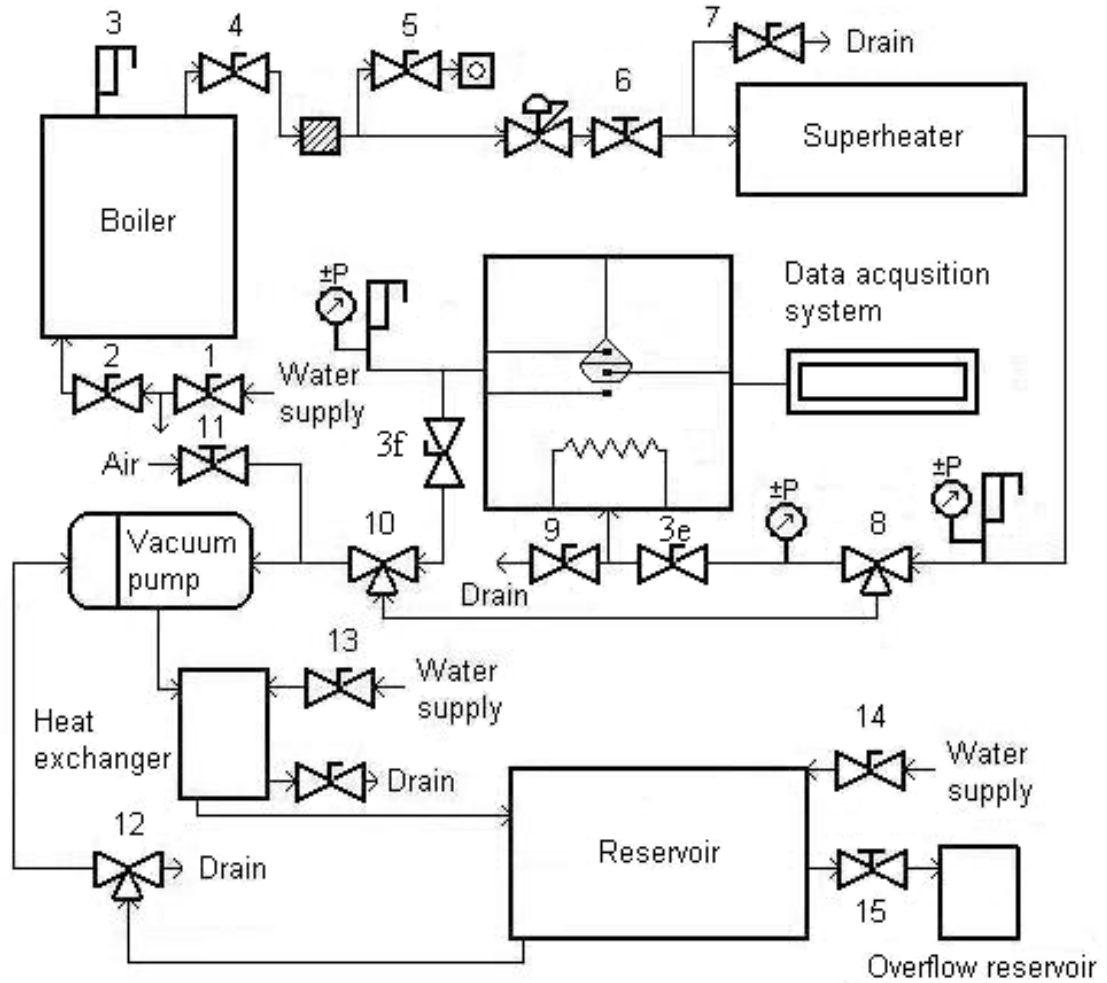


Figure A7. Flow diagram of low pressure superheated steam processing unit.

## **PREPARING THE SUPERHEATED STEAM SYSTEM FOR EXPERIMENTATION**

1. Ensure power bar is turned off and superheater is unplugged.
2. Open VALVE 1 to supply water to the superheated steam system.
3. Open VALVE 2 to fill boiler until water level on indicator is  $\frac{3}{4}$  full.
4. Open VALVE 14 to fill reservoir until water level on indicator is  $\frac{1}{2}$  full.
5. Drain residual water from previous experiment. Ensure containers are situated directly below the pipe outlet located to the left of the superheater and below the drying chamber. Open VALVE 7 to drain water from superheater and VALVE 9 to drain water from drying chamber, close both valves.
6. Turn on power bar and turn dial on boiler to 10 (red light will illuminate). An electric heating tape wrapped around the chamber will begin to heat the chamber.
7. Set superheater to the lowest setting and plug cord into power outlet. Then turn dial to 9.
8. Open VALVE 12 to allow water to flow from the reservoir to vacuum pump.
9. Turn VALVES 8 and 10 to bypass flow from the drying chamber.
10. Ensure VALVE 6 is closed.

Currently, the boiler is set to a maximum pressure of 40 psi. Allow pressure to rise to at least 30 psi before beginning experiment to maintain a steady flow of steam throughout experiment (this will take upwards of 20 min.).

## **CONDUCTING AN EXPERIMENT**

11. Open VALVE 13 to supply water to the heat exchanger.
12. When the boiler pressure is adequate, slowly open VALVE 6 to allow steam to flow through the system.
13. Turn on the vacuum pump.
14. Turn VALVES 8 and 10 to allow superheated steam to flow through the drying chamber.

The initial flow of steam will not be superheated. Allowing steam to run through the system will reduce saturation of the sample. This may take up to 15 min. When all thermocouples show that the chamber has been filled with superheated steam for approximately 3-5 minutes, the sample can be placed into the chamber.

15. Bypass steam from the chamber using VALVES 8 and 10.
16. Place sample in drying chamber. Hook the basket onto the wire suspended from the middle of chamber and fit into the suspended wire ring. Ensure the thermocouple, extending from the wire ring, penetrates the sample from the underside of the basket.
17. Close the door, securing the nuts tightly with a wrench.
18. Re-direct steam to the chamber.

## **PRESSURE**

19. To adjust or maintain a desired pressure inside the drying chamber, either the amount of steam or vacuum applied to the chamber can be adjusted.

i) The amount of steam flowing into the chamber is adjusted by opening or closing VALVE 6.

ii) The amount of vacuum applied to the system is adjusted by opening or closing VALVE 11.

## **TEMPERATURE**

20. To adjust or maintain a desired temperature inside the drying chamber, the amount of steam and temperature of the superheated steam can be adjusted

i) The amount of steam flowing into the chamber is adjusted by opening or closing VALVE 6.

ii) The temperature of the superheated steam is adjusted by increasing or decreasing the setting on the superheater.

## **REMOVING THE SAMPLE MID-EXPERIMENT**

21. Turn VALVES 8 and 10 to bypass flow of superheated stream from the drying chamber.
22. Carefully loosen nuts on the chamber door and remove sample, immediately replacing the door.

## **RE-PLACING THE SAMPLE AND CONTINUING THE EXPERIMENT**

23. Return sample to the drying chamber.
24. Turn VALVES 8 and 10 to re-direct steam into the drying chamber.

## **SHUTTING DOWN THE SUPERHEATED STEAM SYSTEM**

25. Turn off vacuum pump.
26. Immediately turn VALVES 8 and 10 to bypass flow of stream from the drying chamber.
27. Close VALVE 6.
28. Remove sample.
29. Turn dial on boiler down to 0 and turn dial on superheater to lowest setting. Turn



off power bar and un-plug superheater.

30. Turn VALVE 12 to drain Reservoir, turn VALVE 12 once again to drain the vacuum pump.

31. Close VALVES 1, 2 and 13.

NOTE: The water level in the Boiler should not fall below  $\frac{1}{2}$  on its indicator. As the Boiler produces and outputs steam to the system, the water level will gradually decrease. VALVE 2 can be opened to fill the Boiler (will not affect the current operation of the system).

In addition, as steam flows through the system, it will condense and collect in the Reservoir. To prevent overflow, avoid a water level above  $\frac{3}{4}$  on its indicator. When the water level is significant, VALVE 15 can be opened to drain excess water from the Reservoir.

### **MEASURING FLOWRATE**

32. During the set-up, open VALVE 15.

33. Fill reservoir until water flows from VALVE 15. Allow excess water to drain.

34. As steam flows through the system, collect the surplus of water caused by the steam condensate.

35. Measure the volume of water collected. Divide the volume by the duration of time steam was allowed to flow through the system.

## Appendix A.8

### INSTRUCTION FOR DATA ACQUISITION PROGRAM - AGILENT BENCHLINK DATA LOGGER FOR TEMPERATURE READINGS

\* This instruction gives very important step-by-step procedures that must be followed in order to obtain valid readings.

1. Turn on the computer and monitor.
2. Switch on the data acquisition unit and open the Agilent Benchlink Data Logger program located on the desktop.
3. A welcome window as follow will pop-up. Select “Open an existing setup” and click “OK”.

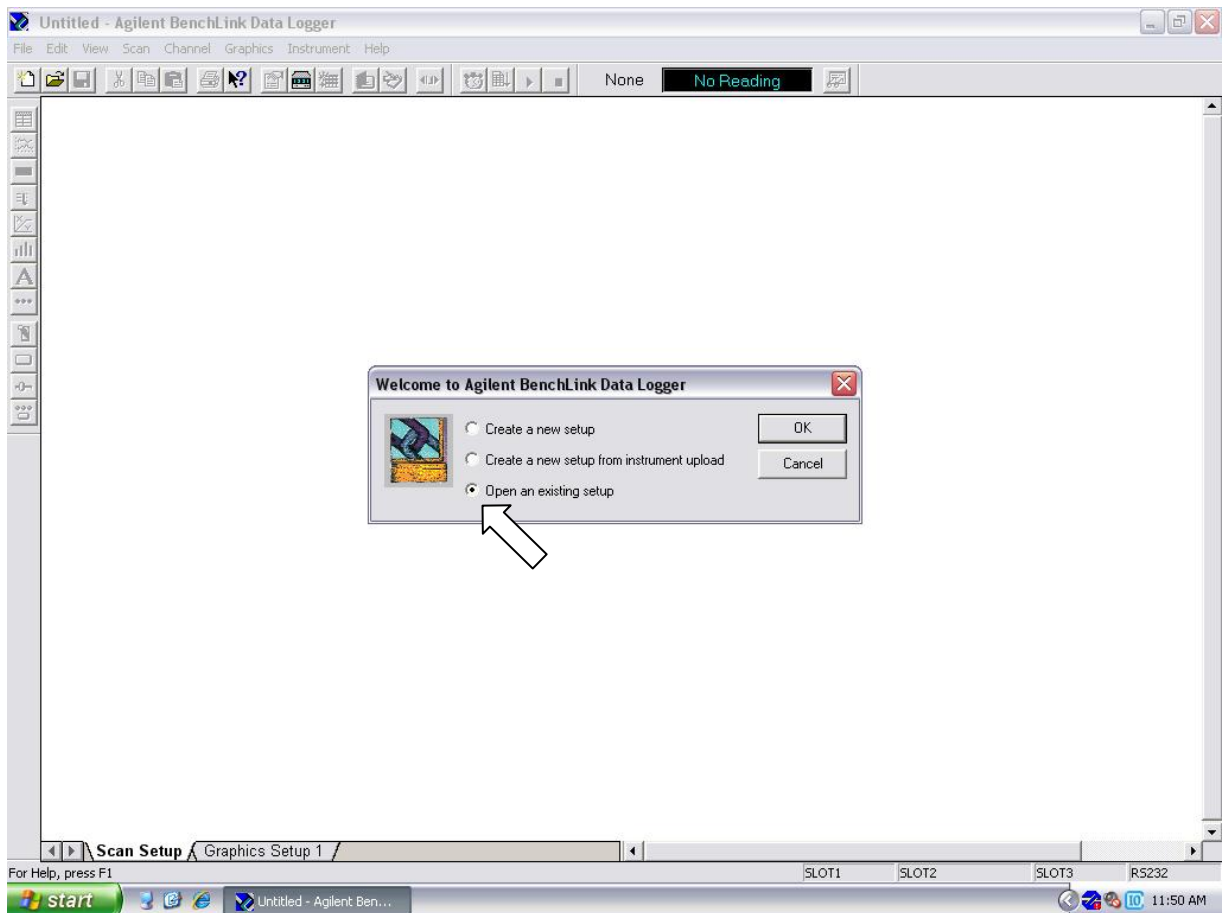
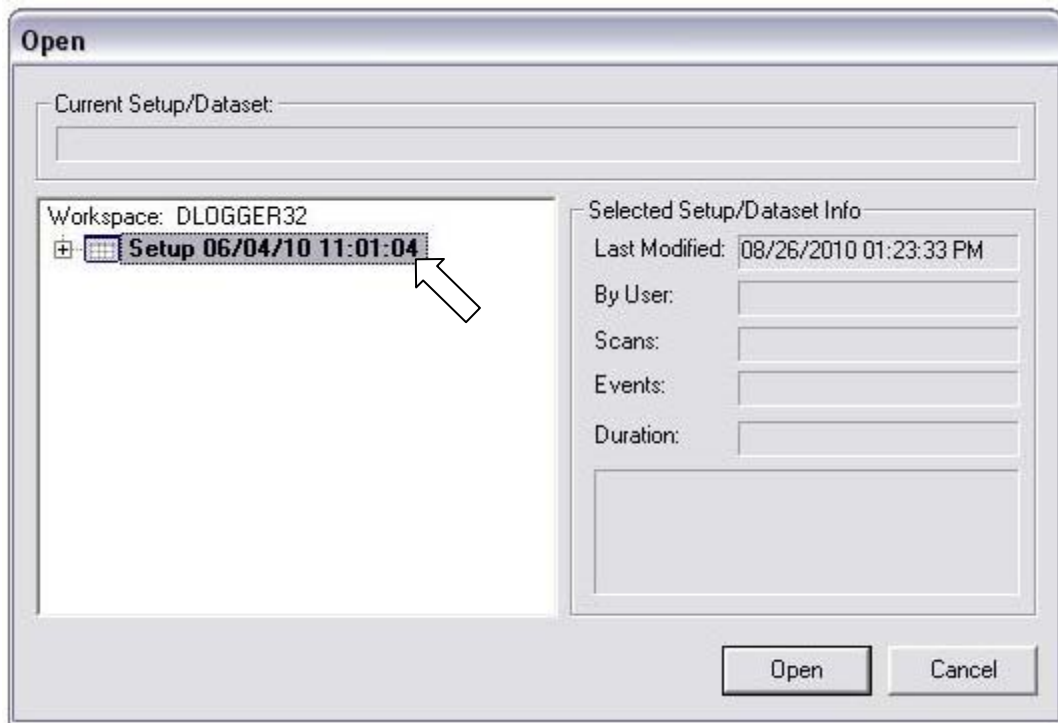


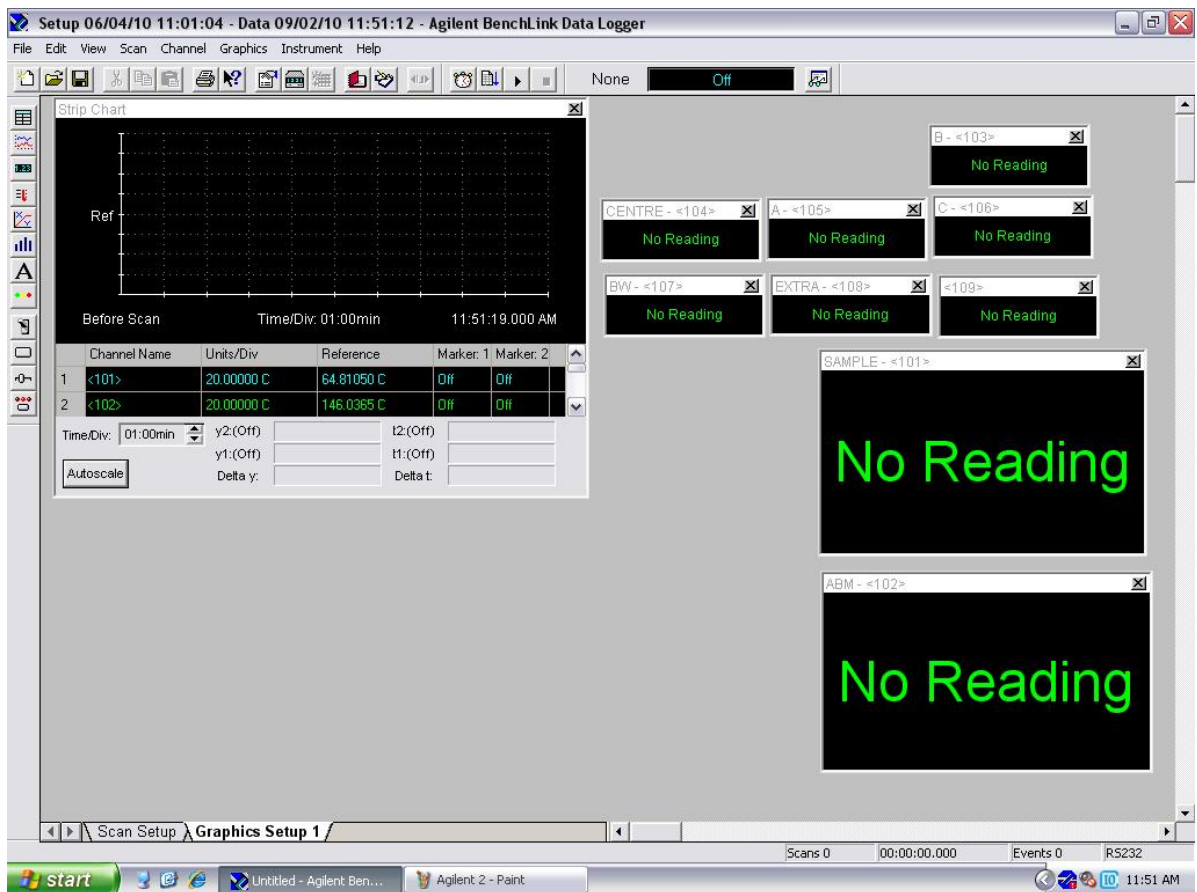
Figure A8-1. Agilent program welcome window.

4. A second window will pop up. Choose the only set up available “Setup 06/04/10 11:01:04” and then click “Open”.



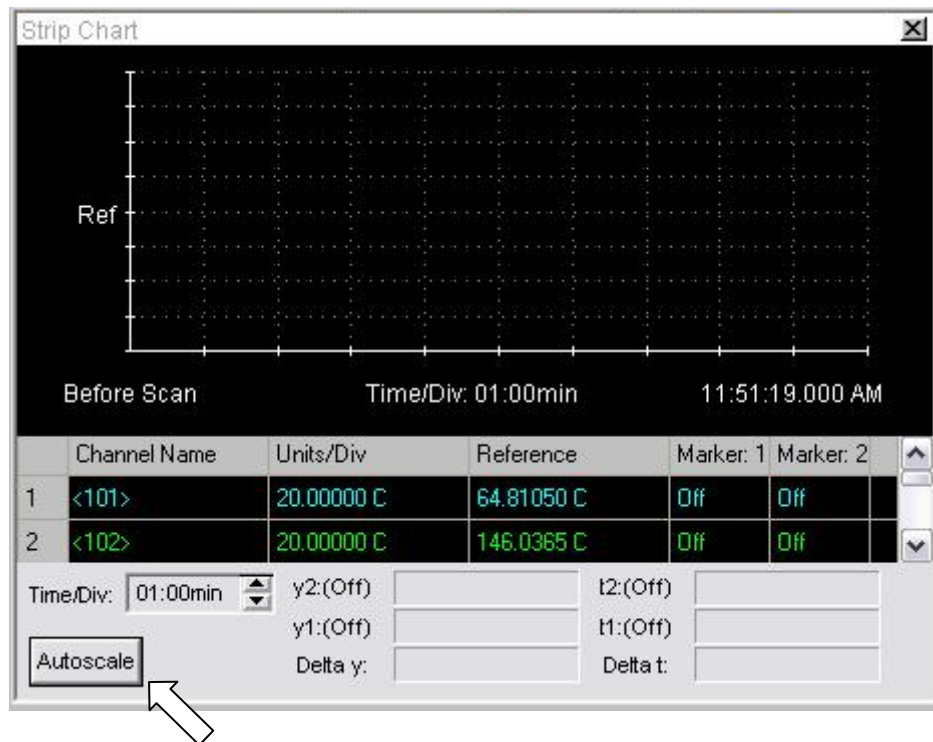
**Figure A8-2. Agilent program setup pop-up window.**

5. A strip chart and nine reading boxes show up. The strip chart shows the trend of the temperature readings from all the nine thermocouples against time. The nine reading boxes show the corresponding thermocouple temperature reading. On the top left corner of each box, there is a name which refers to the corresponding thermocouple. There are two reading boxes that are larger in size than the other seven, they give the two thermocouple readings that are considered the most important (Sample and inlet temperatures).



**Figure A8-3. Agilent program interface.**

6. As temperature readings are constantly recorded and the trend lines are continuously growing, clicking the button “Autoscale” on the strip chart gives the most recent trend of the readings.



**Figure A8-4. Agilent program strip chart.**

7. To acquire data, select the “Start Scan” icon on the toolbar and click “Start” on the following pop-up.

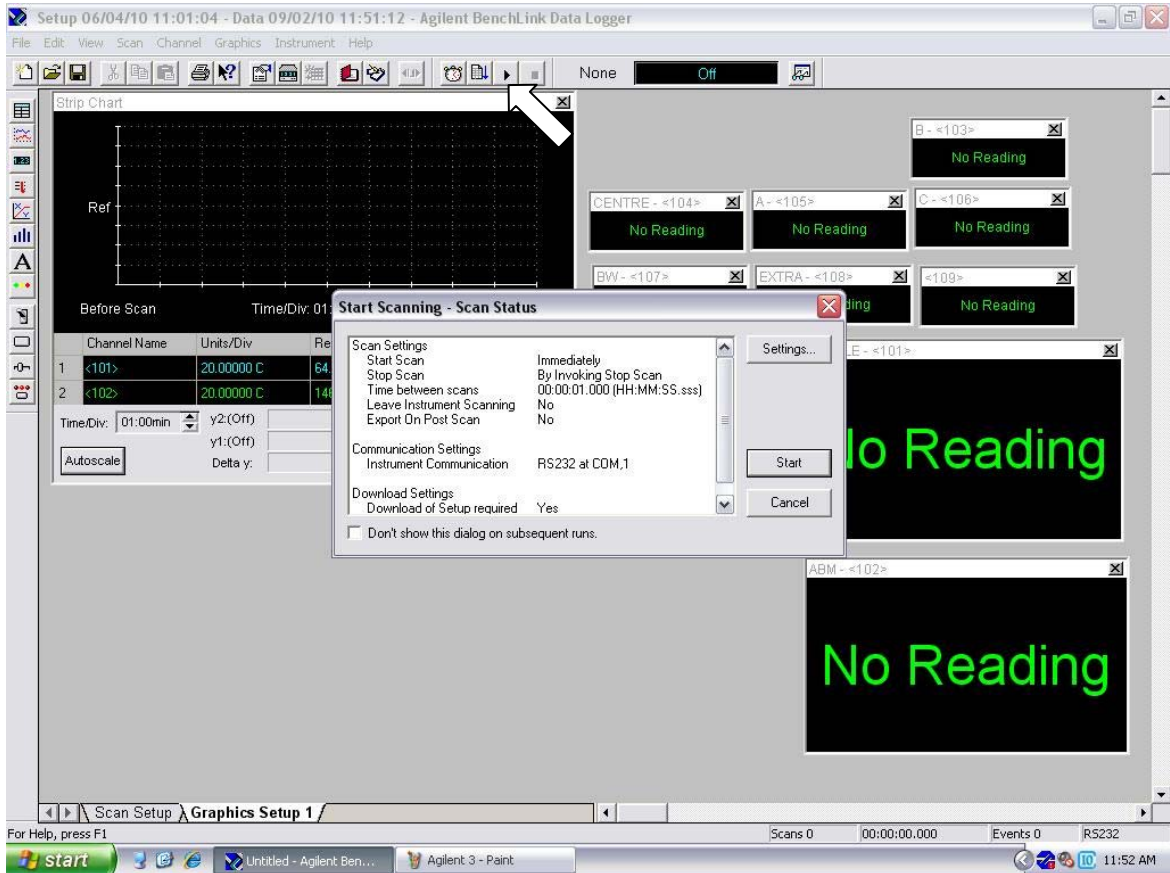


Figure A8-5. Agilent program starting screen.

- To stop data collection, select the “Stop Scan” icon on the toolbar. A window will pop-up, click the “OK” button to save, or click “Delete” to discard the data.

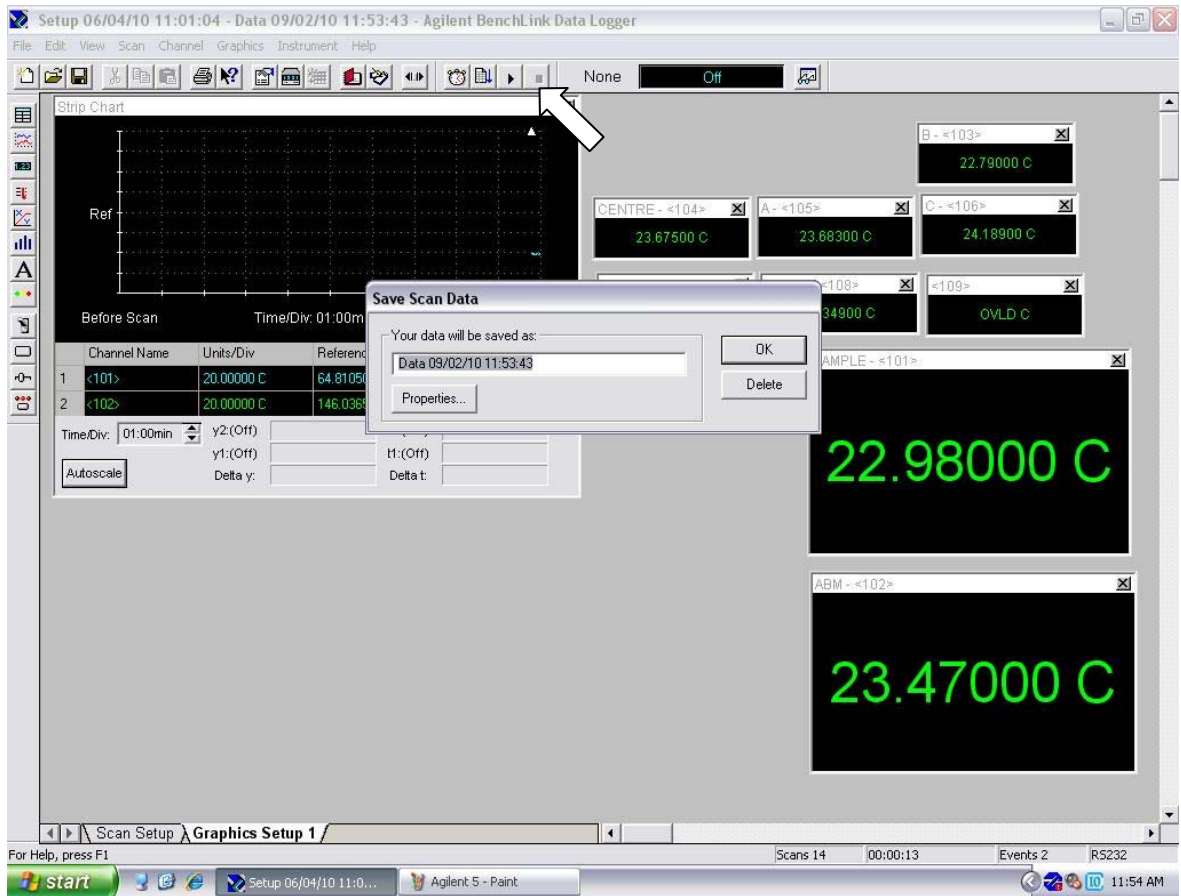
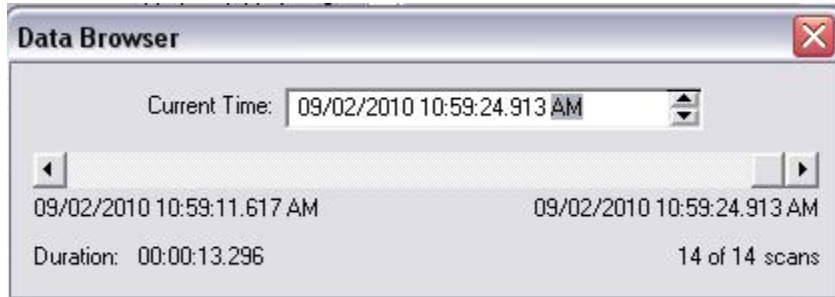


Figure A8-6. Agilent program stopping screen.

9. A browser pop-up will appear to allow for data review. Simply close the browser after review.



**Figure A8-7. Agilent program data browser pop-up window.**



10. When saving data, select “Export Data...” under File.

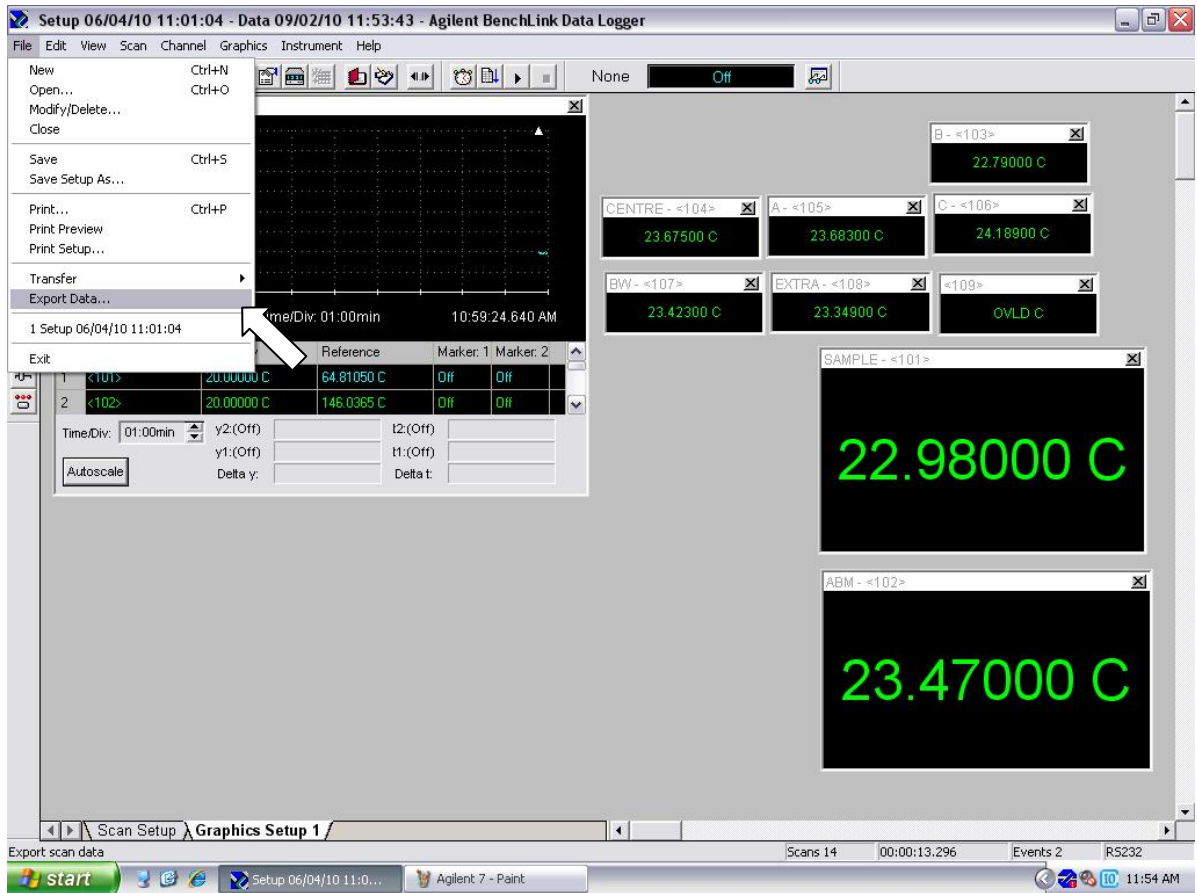


Figure A8-8. Agilent program exporting data screen.

Choose “comma delimited” (CSV) as the output format and rename the file in the file destination box, then click “OK”.

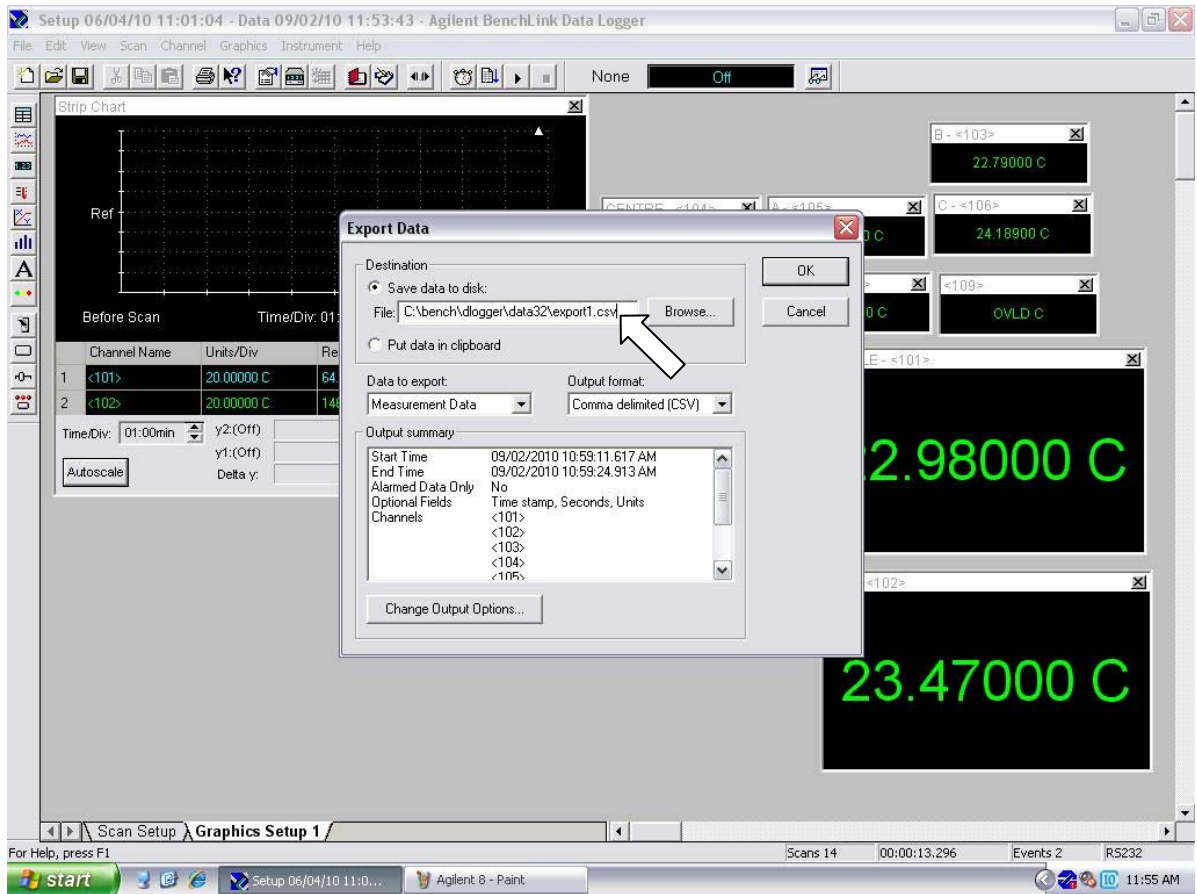


Figure A8-9. Agilent program renaming file screen.

11. After saving the data, simply close the program by clicking the “X” on the top right corner. Please note that it is very important to click “NO” on the pop-up window. This is to keep all the original setup from being set to other values.

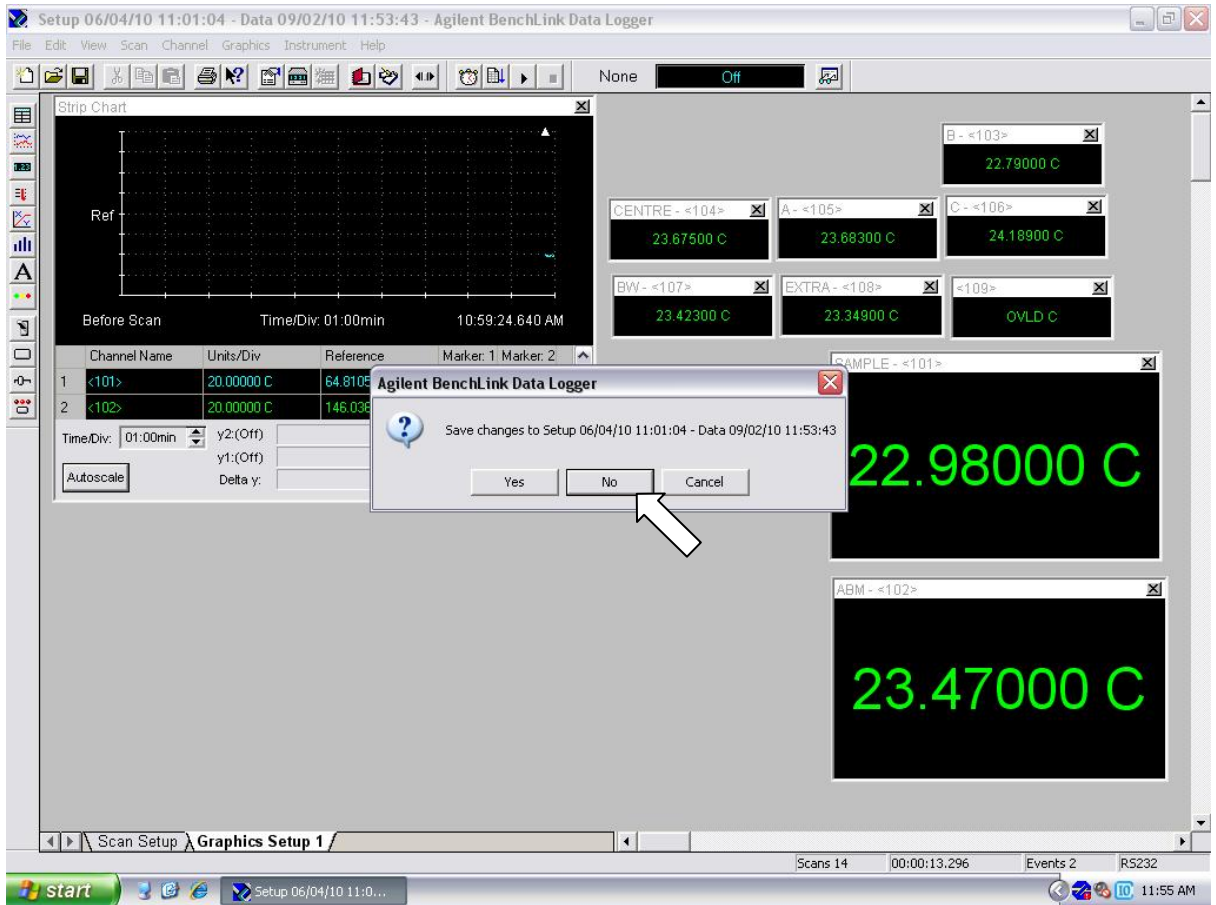


Figure A8-10. Agilent program saving data setup screen.