EFFECTS OF TEMPERATURE CONDITIONS AND HEAT TREATMENT
WITHIN A MULTIPLE EFFECT EVAPORATOR ON THIN STILLAGE
FOULING CHARACTERISTICS

BY

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THESIS

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ABSTRACT

Heat transfer fouling is the accumulation and formation of unwanted materials on heat transfer surfaces which leads to a decrease in the overall heat transfer coefficient. Fouling of heat transfer equipment increases energy consumption and maintenance costs and thus decreases processing efficiency. In the fuel ethanol industry, evaporator fouling occurs when thin stillage is concentrated. Fouling affects the efficiency and environmental footprint of more than 200 biorefineries in the US. Thin stillage is the liquid fraction of unfermented materials from fermentation and is composed of carbohydrate, protein, fat and ash.

Research on thin stillage fouling has focused on effects of corn oil, pH, Reynolds number, solids concentration and carbohydrates (Singh et al 1999, Wilkins et al 2006ab, Arora et al 2010, Challa et al 2014). However, temperature and heat treatment effects on thin still fouling have not been studied. This study investigated the influence of bulk fluid and initial probe temperatures on thin stillage fouling characteristics. Experiments were conducted using model thin stillage (1% starch solution) and commercial thin stillage with varied temperature conditions. We found that temperature had effects on thin stillage fouling. Increase of initial probe temperature would increase fouling rates and maximum fouling resistance for both commercial thin stillage and model thin stillage. At an initial probe temperature of 120°C, higher bulk temperature (80°C) increased fouling rates and reduced induction periods.

Effects of evaporator heat treatment were studied by examining fouling behavior among samples from different locations of a multiple effect evaporator. Samples before and after plant cleaning were collected to study effects of plant cleaning. Effects of heat treatment within an evaporator were not detected. Fouling tendencies were reduced after plant cleaning.
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# TABLE OF CONTENTS

CHAPTER 1. INTRODUCTION AND OBJECTIVES ................................................................. 1
  1.1. Introduction .............................................................................................................. 1
  1.2. Objectives ............................................................................................................... 3

CHAPTER 2. LITERATURE REVIEW ...................................................................................... 4
  2.1. Introduction .............................................................................................................. 4
  2.2. Classification of fouling ........................................................................................... 5
  2.3. Progression of fouling .............................................................................................. 6
  2.4. Factors affecting fouling .......................................................................................... 6
  2.5. Fouling measurements and monitoring .................................................................... 8
  2.6. Fouling in the corn ethanol industry ....................................................................... 14
  2.7. Fouling in the oil refinery industry ......................................................................... 23
  2.8. Fouling in the dairy industry ................................................................................... 25

CHAPTER 3. SURFACE AND BULK TEMPERATURE EFFECTS ON FOULING
  CHARACTERISTICS OF THIN STILLAGE ......................................................................... 27
  3.1. Introduction and objectives ..................................................................................... 27
  3.2. Methods and materials ............................................................................................ 28
  3.3. Results and discussion ............................................................................................. 37
  3.4. Conclusions .............................................................................................................. 47

CHAPTER 4. EVAPORATOR HEAT TREATMENT EFFECTS ON FOULING
  CHARACTERISTICS OF THIN STILLAGE ......................................................................... 49
  4.1. Introduction and objectives ..................................................................................... 49
  4.2. Materials and methods .............................................................................................. 50
  4.3. Results and discussion ............................................................................................. 55
  4.4. Conclusions .............................................................................................................. 66

CHAPTER 5. RECOMMENDATIONS ..................................................................................... 68

LITERATURE CITED: ........................................................................................................... 69

APPENDIX A. FOULING TEST PROCEDURE .................................................................. 73

APPENDIX B. FOULING APPARATUS .............................................................................. 75
CHAPTER 1. INTRODUCTION AND OBJECTIVES

1.1. Introduction

The US Clean Air Act (1990) was established for reformulated gasoline to reduce air pollution. Ethanol and methyl tertiary butyl ether (MTBE) were approved as oxygenates and fuel additives. However, as the Environmental Protection Agency (EPA) phased out MTBE because of environmental and human health issues, ethanol became the only suitable fuel additive in the market. As a result, ethanol demand increased and ethanol production increased more than 10 times during the past 10 years, from 1.3 billion gal in 1994 to 14.3 billion gal in 2014 (RFA 2015). In 2015, there were more than 200 ethanol plants in the US.

Fuel ethanol is made from corn by either dry grind or wet milling. In 2015, 90% of ethanol was produced from the dry grind process (RFA 2015). In the dry grind process, nonfermentables (whole stillage) are centrifuged to separate soluble solids from insoluble solids. Thin stillage, the overflow from the centrifuge, is concentrated using multiple effect evaporators from 6 to 30% total solids to form a process stream known as syrup (condensed distillers solubles) which is mixed with wet grains and dried further to produce a coproduct called distillers dried grains with solubles (DDGS).

Heat transfer fouling is the accumulation and formation of unwanted materials on heat transfer surfaces. This impedes heat transfer and increases resistance to fluid flow. Fouling affects energy consumption of industrial processes and also results in frequent shut down and cleaning. There are five types of fouling mechanisms (Bott 1995), which makes fouling a complicated phenomenon. Awad (2011) estimated heat exchanger fouling costs consumed 0.25%
of the US gross national product ($14.2 Billion).

Heat transfer fouling increases capital investment to compensate for the reduced rate of heat transfer as well as increased operating costs to maintain desired temperatures and fluid conditions. Maintenance costs are increased to remedy effects of fouling (Bott 2007). Fouling of heat exchangers may cause environmental hazards and emissions (Muller-Steinhagen et al 2009).

In the dairy industry, fouling reduces heat transfer efficiency, increases pumping power requirements and affects the economics of processing plants (Toyoda et al 1994). Milk fouling is rapid and heat exchangers need to be cleaned on a daily basis (Bansal and Chen 2006a).

Milk fouling has been studied extensively in terms of milk composition, operating conditions in heat exchangers, type and characteristics of heat exchangers, presence of microorganisms and fouling location.

In the corn dry grind process, fouling in evaporators provides resistance to heat transfer and restricts the flow of thin stillage. Fouling deposits must be removed periodically from the heat transfer surface. Periodical cleaning and maintenance results in increased capital, operation and maintenance costs. Understanding fouling tendencies would result in reduced labor costs, downtime and cleaning chemical costs (Agbisit et al 2003; Wilkins et al 2006b; Arora et al 2010).

Previous studies on thin stillage fouling included effects of Reynolds number, pH and membrane filtration as well as oil, carbohydrate and total solids contents (Singh et al 1999; Agbisit et al 2003; Wilkins et al 2006a; Wilkins et al 2006b; Arora et al 2010; Rausch et al 2013; Challa et al 2015). Among these studies, bulk temperatures varied from 40 to 75°C and initial probe temperatures varied from 100 to 120°C.

Because of the complex compositions and variation of commercial thin stillage, Rausch et
al (2013) developed model fluids to study thin stillage fouling. Challa (2014) examined fouling characteristics of thin stillage and concentrates from different locations of a multiple effect evaporator in a dry grind facility. Total solids of the sample varied from 7 to 11%. Fouling rate increased with the increase of sample solids concentration.

Studies of interface temperatures between fouling liquid and the heat transfer surface were conducted. Effects of initial probe temperatures and bulk temperatures on thin stillage fouling characteristics were examined. This will provide a foundation for future work on operating annular probe apparatuses with commercial thin stillage samples and model thin stillage. Effects of heat treatment on thin stillage fouling characteristics of a multiple effect evaporator also were studied. Samples before and after plant cleaning were collected to study effects of plant cleaning.

1.2. Objectives

The objectives of this study were to:

1. Determine effects of bulk temperature and initial probe temperature of the test apparatus on thin stillage fouling characteristics.

2. Observe effects of exposure to evaporator heat treatment and dry grind facility shut down on fouling.
CHAPTER 2. LITERATURE REVIEW

2.1. Introduction

Heat transfer fouling is the accumulation of substances on the surface interface during a heat transfer process. Presence of these deposits decrease thermal conductivity and their resistance to heat transfer deteriorates the capacity or efficiency of the transfer heat surface. Additionally, accumulation of deposits on the heat transfer surfaces reduces cross section area, increasing pressure requirement for the exchanger (Bott 1995; Awad 2011).

Fouling is a complex phenomenon in which chemical, biological, solubility and corrosion processes may occur. Fouling may be involved in interfaces such as gas-liquid, liquid-liquid, gas-solid and liquid-solid (Epstein 1983; Bott 1995). Fouling of heat transfer surfaces is one of the most important problems in heat transfer equipment and affects most process industries, including oil refining, pulp and paper manufacturing, polymer and fiber production and food and bioprocessing industries (Awad 2011).

Overall annular cost of fouling was estimated to be 0.3% of the UK gross national product (Epstein 1983). Bott (1995) estimated the annual cost of fouling in the US to be 15 to 20 billion in 1993. Coletti and Hewitt (2015) estimated fouling related costs in crude oil refineries were $3.6 billion in 2014. There are no published reports of fouling costs for the corn processing industry. Costs associated with heat transfer fouling include the following (Epstein 1983; Bott 1995):

1. Increased capital investment due to oversized heat transfer equipment to accommodate fouling effects.
2. Additional operation costs; energy losses due to thermal inefficiencies and increased pressure requirement.

3. Maintenance costs, including cleaning of heat exchangers and use of antifoulants.

4. Loss of production due to interruptions of normal operation as well as losses of product quality due to fouling.

2.2. Classification of fouling

Fouling is a complex phenomenon that involves physical, chemical and biological processes. Fouling can be classified into five types (Epstein 1983; Awad 2011):

1. Precipitation fouling: fouling (scaling) which is crystallization from solution of dissolved substances onto the heat transfer surface, and solidification, freezing of a pure liquid onto subcooled surfaces.

2. Particulate fouling: the accumulation of suspended particles in the process fluid onto the heat transfer surfaces.

3. Biological fouling: the attachment and growth of macroorganisms and/or microorganisms to heat transfer surfaces.

4. Chemical reaction fouling: deposit formation at the heat transfer surface by chemical reactions in which the surface itself is not a reactant.

5. Corrosion fouling: the accumulation of indigenous corrosion products, which involves chemical or electrochemical reaction between the heat transfer surface itself and the fluid stream, on the heat transfer surfaces.
2.3. Progression of fouling

The general fouling progress is considered to be the net result of two simultaneous processes; a deposition process and a removal process. Five basic stages may be used to describe the sequence of fouling (Epstein 1983; Awad 2011):

1. **Initiation:** also known as induction, delay, incubation or surface conditioning. A time that foulant materials form in the bulk of fluid and are not yet transported to the heat transfer surface.

2. **Transport (mass transfer):** the transport of foulant materials to the deposit fluid surface.

3. **Attachment (adhesion):** bonding of foulant on the heat transfer surface that leads to deposit formation.

4. **Removal (detachment):** the release of fouling deposit caused by randomly distributed turbulence bursts, shear forces, resolution and erosion.

5. **Aging:** can decrease or increase the strength of deposit while changes in crystal and chemical structure of deposit may take place.

The third stage (attachment) which initiates fouling deposition on the heat transfer surface can be the crucial part to fouling, yet is not well understood. Surface conditions, surface forces and sticking probability are three key factors affecting the attachment process (Awad 2011).

2.4. Factors affecting fouling

Previous fouling research (Changani et al 1997; Bansal and Chen 2006a; Kazi 2012; Sadeghinezhad et al 2015) showed that factors affecting fouling are composition and concentrations of test fluid, operating conditions of experiments and types and properties of heat exchangers used in the operation.
2.4.1. Composition and concentration

According to Awad (2011), fluid viscosity and density play important roles in fouling. Fluid viscosity may influence sublayer thickness where deposition processes take place. Both viscosity and density influenced shear stress which in turn affected fouling.

Effects of single or multiple compounds on fouling behavior were determined (Dausin et al 1987; Singh et al 1999; Bansal and Chen 2006a). Agbisit et al (2003) and Arora et al (2010) studied effects of solids concentration on fouling in corn processing using membrane filtration and observed positive correlations among fouling rates and solids concentrations.

2.4.2. Operating conditions

Fluid flow velocity affects fouling as velocity has a direct influence on deposition and removal rates (Awad 2011; Kazi 2012). In most cases, increased flow rate leads to increased shear stress and would result in decreases fouling rates. Reynolds number also was found to have a similar influence on fouling rate (Belmar-Beiny et al 1993; Chen and Jebson 1997; Wilkins et al 2006a).

Higher bulk temperature may increase fouling rate (Belmar-Beiny et al 1993; Bansal and Chen 2006a; Kukulka and Devgun 2007; Awad 2011). Surface temperature may have varied effects on fouling depending on fouling categories. For biological and corrosion fouling, increase of surface temperature tends to decrease fouling rates; for crystallization, chemical reaction and particulate fouling, surface temperature could have an increasing effect (Muller-Steinhagen 2011). Effects of pH and other operating conditions also have been studied. (Bansal and Chen 2006a; Wilkins et al 2006b).
2.4.3. Types and properties of heat exchangers

Surface material, surface structure (roughness) of heat exchangers and their types and geometry affect fouling. Surface material is critical to corrosion fouling for the potential of surface to react and form corrosion products. Materials that are noncorrosive and have high thermal conductivity would help to minimize fouling in evaporators (Awad 2011). Kazi (2012) found surface roughness had an effect on the initial fouling rate and scale formation. The rougher surface resulted in higher deposition rates. The design of heat exchangers can reduce or increase fouling. Awad (2011) concluded two effects of surface roughness on fouling: the provision of “nucleation sites” that promote the setting down of the initial deposits and the turbulence effects within the flowing fluid. A better surface finish delayed the process of fouling on the surface while rough surfaces increased particulate deposition.

2.5. Fouling measurements and monitoring

Fouling characteristics can be measured experimentally both qualitatively and quantitatively (Awad 2011):

1. Direct weighing: a simple method for assessing the extent of deposition on test surfaces. An accurate weighing method and a sensitive balance are important to reduce measuring error.

2. Thickness measurement: a straightforward method to measure the extent of fouling. Direct measurement is difficult as the layer of deposit is thin; removing the deposit may be needed.

3. Heat transfer measurements: where fouling resistance can determined by changes in overall heat transfer coefficient.
4. Pressure drop: an alternative method to direct heat transfer measurement. Pressure drop is affected by the properties of deposits.

More and more techniques are developed to better monitor fouling without an interruption of operating process. For example, laser technique can be used to examine the accumulation and removal of deposits. Microscopic analyses of deposits can provide useful information to explore the mechanisms of fouling (Awad 2011).

2.5.1. Heat transfer resistance

Heat transfer fouling results in a thermally insulating layer on the surface. The various resistances to heat transfer confronted as heat flow from a hot fluid to a cold fluid with temperature drops are shown in Fig 2.1. Heat transfer resistances ($R_f$) are in series and overall heat transfer coefficient ($U$) is defined as:

$$\frac{1}{U} = \sum R_f$$

In the clean heat exchanger (Fig. 2.1):

$$\frac{1}{U_c} = R_1 + R_W + R_2$$

Where $U_c$ is the overall heat transfer coefficient of cleaned surface, $R_1$ and $R_2$ are the individual heat transfer surfaces on the hot and cold sides of the heated wall and $R_W$ is the individual heat transfer resistance from the wall.

In the fouled surface (Fig. 2.1):
Figure 2.1. Heat transfer resistance and temperature profiles (Somerscales and Knudsen 1981)

\[
\frac{1}{U_f} = R_1 + R_{f1} + R_W + R_{f2} + R_2
\]

Where \(U_f\) is the overall heat transfer coefficient of fouled surface; \(R_{f1}\) and \(R_{f2}\) are individual heat transfer resistances from fouling on hot and cold sides of the wall.

Fouling resistances \((R_f)\) of deposits on both sides of the wall can be determined from the differences of the overall heat transfer coefficients of fouled and clean surfaces:

\[
R_f = \frac{1}{U_f} - \frac{1}{U_c} = R_{f1} + R_{f2}
\]
2.5.2. Fouling apparatus

Because of difficulties in obtaining fouling data (fluctuations in fluid compositions and operating conditions) from heat exchangers, several experimental test sections have been developed to acquire fouling parameters (Somerscales and Knudsen 1981). Circular, annular, spherical geometry test sections; electrically heated wires and coils and pilot scale heat exchangers are examples of fouling test apparatuses.

A tubular fouling unit (TFU) was used in fouling studies (Belmar-Beiny et al 1993; Wilson and Watkinson 1996). TFU was heated by direct passage of electrical current and could be cut open to measure deposit mass and structure (Wilson and Watkinson 1996); they used TFU to measure both pressure drop and heat transfer resistance.

The annular geometry test section (Fig. 2.2), has been used in several fouling studies (Panchal and Watkinson 1993; Wilson and Watkinson 1996; Agbisit et al 2003; Wilkins et al 2006a; Wilkins et al 2006b; Arora et al 2010; Rausch et al 2013; Challa et al 2015).

Figure 2.2. Annular geometry test section
The annular test unit consists of an outer cylinder with a metal probe in the center (Fig. 2.2). Fluid flows in the annular space between the probe and outer tube section. A small section of the probe is heated with an electric resistance heater and thermocouples are embedded in the inner wall of the probe to monitor wall temperature changes caused by accumulation of fouling deposits. A fouling test is operated at constant heat flux, velocity and bulk temperature.

Using the surface temperatures measured by the thermocouples, the overall heat transfer coefficient (U) can be determined from its definition:

\[ U = \frac{q/A}{T_s - T_b} \]

Where \( q/A \), defined as thermal power per unit area, is the amount of heat transferred (heat flux); \( T_s \) is the probe surface temperature and \( T_b \) is the fluid bulk temperature. \( T_s \) can be calculated from the inner wall temperature:

\[ T_s = T_w - \left( \frac{x}{k} \right) \left( \frac{q}{A} \right) \]

Where \( x \) is the distance from the thermocouple to the probe surface and \( k \) is thermal conductivity of the probe metal. The \( x/k \) value is calculated using the method of Wilson (1915), where a linear plot of \( 1/U \) vs \( V^n \) (\( U \) is the overall heat transfer coefficient and \( V \) is the fluid velocity) is drawn using experimental data (Fig. 2.3). The value of \( n \) is determined empirically until the plot is a linear line for an intercept of \( R_w \) (thermal resistance from wall surface). Fouling resistance at time \( t \) (\( R_{ft} \)) can be determined by heat transfer coefficients:

\[ R_{ft} = \frac{1}{U_t} - \frac{1}{U_0} \]
Where $U_t$ (kW/m$^2$K) is the overall heat transfer coefficient at time $t$, $U_0$ is the initial (t=0) overall heat transfer coefficient at the beginning of the fouling test. Therefore, by monitoring the bulk and inner wall temperatures, fouling resistance at each time point can be calculated. Fouling curves can be derived from the data (see section 2.5.3).

![Figure 2.3. Sketch of Wilson plot](image)

### 2.5.3. Fouling curve

The overall process of fouling can be described by $R_f$ (fouling resistance) measured by a test section. The presentation of various modes of fouling with regard to time is known as a fouling curve (Awad 2011). A typical process of fouling in heat transfer equipment has been summarized in three mode curves shown in Fig. 2.4 (Kazi 2012).

A linear fouling curve (A) indicates constant deposit and removal rates in the process. A falling rate fouling (B) is where fouling resistance increases with time but deposit and removal rates are not constant. In asymptotic fouling (C), constant deposit and removal rates are proportional to the deposit thickness until both rates are equal. When this steady state is reached,
there is no net increase of fouling deposits. Asymptotic fouling is the most important mode as it exists widely in industrial applications (Epstein 1983; Awad 2011).

Initiation or induction periods are considered where there is no appreciable deposit on the heat exchanger surfaces. The initial growth of deposits may lead to an increase in the heat transfer coefficient resulting a negative fouling resistance (Fig. 2.4). Initial periods vary and are difficult to estimate; many mathematical models ignore this period of time (Bott 1995).

Figure 2.4. Fouling resistance vs time (Kazi 2012)

2.6. Fouling in the corn ethanol industry

2.6.1. Maize dry grind and wet milling processes

Corn is composed largely of starch which can be hydrolyzed into sugars and fermented to ethanol using yeast. Commercial production of corn ethanol uses either a dry grind or a wet
milling process. These processes differ with respect to complexity and associated capital costs, numbers and types of coproducts produced. Also there is flexibility to produce different kinds of primary products. Dry grind is the most prevalent process (90%) and much of the current industry uses this technology as it is a simpler process with lower capital costs relative to wet milling and produces higher ethanol yields (2.7 to 2.8 gal/bu corn).

In the dry grind process (Fig. 2.5), whole corn is ground, slurried in water and cooked with \(\alpha\)-amylase enzymes to hydrolyze starch into dextrins during liquefaction. The dextrins are hydrolyzed into glucose by glucoamylase and converted to ethanol by yeast during simultaneous saccharification and fermentation (SSF). Ethanol is separated by distillation from nonfermentables (whole stillage) and water to a concentration of 95% (190 proof). Additional water is removed by molecular sieves to produce 100% (200 proof) ethanol. Whole stillage, which contains fiber, oil, protein and other unfermented components of the grain and yeast cells, is centrifuged to separate soluble solids from insoluble solids.

![Figure 2.5. A schematic of conventional dry grind process](image)

Figure 2.5. A schematic of conventional dry grind process
Thin stillage, the over flow from the centrifuge, is concentrated using multiple effects evaporators to 30% total solids contents (distillers solubles or syrup) (Singh et al 1999), while 15% of thin stillage is recycled as process water which goes back to slurry (Kwiatkowski et al 2006). Insolubles from the centrifuge (wet grains or distillers grains) and syrup are mixed and dried to produce distillers dried grains with solubles (DDGS), the main coproduct in the corn dry grind industry. In thin stillage evaporation, rapid fouling takes place which leads to periodic shutdowns for cleaning and maintenance (Singh et al 1999; Wilkins et al 2006a).

Wet milling is a process in which corn is fractionated into four components (starch, germ, fiber and protein). The starch recovered can be used to make products such as high fructose corn syrup, ethanol or chemicals. Steeping, germ recovery, fiber recovery, protein recovery and starch washing are the five basic processing steps in wet milling. A schematic of the wet milling process is shown in Fig. 2.6.

According to Rausch et al (2005), steeping is the first and crucial step of wet milling as steeping improves the separation of kernel components and influences starch quality and quantity and coproduct characteristics. Light steepwater (4 to 8% solids contents), the material remaining after steeping, is concentrated to heavy steepwater (35 to 40% solids contents) by multiple effects evaporators. Steepwater consists of proteins, carbohydrates, fat and inorganics which can form deposits on heat transfer surfaces during the evaporation process (Agbisit et al 2003).
Figure 2.6. A schematic of the corn wet milling process (Rausch et al 2005)
2.6.2. Evaporation in the corn ethanol industry

The objective of evaporation is to concentrate a solution consisting of a nonvolatile solute and a volatile solvent (Smith 2001). Evaporation is conducted by vaporizing a portion of the solvent to produce a concentrated solution of thick liquor which could be a valuable product. Evaporation process is affected by characters of the liquid to be concentrated. According to the properties of liquor (e.g., foaming, temperature sensitive, easy to scale) process evaporators are designed.

Evaporators can be categorized into two types; long tube vertical and agitated film. Tubular exchanger and vapor separator are essential parts of a long tube evaporator. Depending on the material of input liquor, evaporator can be operated as once through or as circulation units. Once through evaporation is used for heat sensitive materials and the feed liquor passes through tube only once, minimizing heat exposure time. Agitated, falling and climbing film evaporators can function in this way (Smith 2001).

Falling film evaporator (Fig. 2.7) can operate at low temperature differences with high heat transfer coefficients and minimal heat exposure time for liquids and thus are used when processing heat sensitive liquids in the food processing industry (Salvagnini and Taqueda 2004). In the falling film evaporator, liquids enters at the top, distributes uniformly before flowing downstream inside the heated tube as a film, and leaves at the bottom (Smith 2001). Vapor developed from the liquids is carried downward with the liquids and leaves from the bottom of the units (Fig. 2.7). A short amount of residence time is achieved due to film flow and continuous operation.
In the dry grind process, stillage evaporation is used to remove excess water from thin stillage. Thin stillage, which contains 5 to 10% total solids, is concentrated to produce syrup, which contains 30 to 50% total solids (Singh et al 1999). In the wet milling process, light steepwater (5 to 8% total solids) is concentrated to heavy steepwater (45 to 50% total solids) through evaporation (Agbisit et al 2003).

Fig. 2.8 is an example of a four effect evaporator. Additional evaporator effects could remove more water per unit of steam supplied, thus improving evaporation system efficiency. Factors like minimal practical condensing temperature of final vapor, maximum practical
product side temperature and cleaning frequency, need to be taken into consideration when designing a multiple effect evaporator. To increase evaporator efficiency, vapor compression is added to the system by recycling vapor from later effects to previous effects. Thermocompression or mechanical compression devices are used to increase the pressure of the vapor (Meredith 2003).

Figure 2.8. Four-effect evaporator (Meredith 2003).
2.6.3. Fouling studies in the corn processing industry

In the corn dry grind industry, thermal energy required for thin stillage evaporation and DDGS drying accounts for 40 to 45% of the total thermal energy and 30 to 40% of total electrical energy (Meredith 2003). During the evaporation process, thin stillage tends to foul rapidly; therefore, periodically cleaning and maintenance is needed (Singh et al 1999). In the corn wet milling process, light steep water (LSW) is concentrated to 35 to 40% heavy steep water by evaporators. Agbisit et al (2003) mentioned that steepwater evaporation is one of the most capital and energy intensive unit operations for the wet milling process, consuming 20% of total energy use and 20% of capital cost. Fouling of steepwater reduces the rate of heat transfer and leads to a regular cleaning, which increases energy and maintenance costs.

An annular fouling test section (described in previous sections) was used in all these studies to measure fouling resistance (Singh et al 1999; Agbisit et al 2003; Wilkins et al 2006a; Wilkins et al 2006b; Arora et al 2010; Rausch et al 2013; Challa et al 2015). Fouling resistance, maximum fouling resistance, fouling rate (derived from the slope of linear regression line of fouling resistance over time), and induction periods are parameters used to quantify fouling characteristics. Depicted in Table 2.1 are operating conditions of previous fouling studies. (Wilkins et al 2006a) studied effects of Reynolds number (Re) on thin stillage fouling. Three batches of thin stillage samples from dry grind facilities were tested at two flow rates (Re = 440 and Re = 880). Fouling rate decreased and induction period increased with the increase of Re, indicating higher velocity and sheer force would reduce fouling. Greater fouling rates and shorter induction periods were observed at pH 3.5 compared with pH 4 or 4.5 (Wilkins et al 2006b).
Table 2.1. Operating conditions in previous fouling studies of corn processing.

<table>
<thead>
<tr>
<th></th>
<th>$T_b$ (^1) (°C)</th>
<th>$T_i$ (^2) (°C)</th>
<th>Sample volume (L)</th>
<th>Test periods (hr)</th>
<th>Flow rate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Singh et al 1999</td>
<td>40 ± 2.5</td>
<td>NA</td>
<td>30</td>
<td>12 to 13</td>
<td>0.26 m/sec</td>
</tr>
<tr>
<td>Agbisit et al 2003</td>
<td>40 ± 1</td>
<td>99 ± 1</td>
<td>30</td>
<td>12</td>
<td>13 ± 0.5 L/min</td>
</tr>
<tr>
<td>Wilkins et al 2006a</td>
<td>40 ± 2</td>
<td>100</td>
<td>30</td>
<td>12</td>
<td>11.3 L/min</td>
</tr>
<tr>
<td></td>
<td>50 ± 2</td>
<td></td>
<td></td>
<td></td>
<td>22.6 L/min</td>
</tr>
<tr>
<td>Wilkins et al 2006b</td>
<td>40 ± 2</td>
<td>100</td>
<td>30</td>
<td>8</td>
<td>5.2 m/sec</td>
</tr>
<tr>
<td>Arora et al 2010</td>
<td>60 ± 2</td>
<td>100</td>
<td>30</td>
<td>22</td>
<td>NA</td>
</tr>
<tr>
<td>Rausch et al 2013</td>
<td>50 ± 2</td>
<td>100</td>
<td>50</td>
<td>10</td>
<td>15.1 L/min</td>
</tr>
<tr>
<td>Challa et al 2015</td>
<td>75 ± 2</td>
<td>120 ± 2</td>
<td>7</td>
<td>5</td>
<td>0.32 to 0.45 m/sec</td>
</tr>
</tbody>
</table>

\(^1\) $T_b$ = bulk temperature.  
\(^2\) $T_i$ = Initial probe temperature

Compositions of thin stillage from different studies are shown in Table 2.2. Singh et al (1999) examined effects of refined corn oil on thin stillage fouling. Four different amounts of refined corn oil were added to thin stillage samples from wet milling and dry grind ethanol plants. Adding oil increased fouling resistance of thin stillage from wet milling plants up to a certain level; it decreased fouling resistances of thin stillage from dry grind plants. Agbisit et al (2003) studied fouling characteristic of raw light steep water (LSW) and membrane filtered steepwater (FSW) and found that microfiltration decreased fouling. Microfiltration treated thin stillage with lower solids contents and changed compositions had lower fouling rates and fouling resistances and longer induction periods compared to unfiltered thin stillage (Arora et al 2010).
Table 2.2. Thin stillage compositions from various researchers.

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Dry matter</td>
<td>5.0 (%db)</td>
<td>4.4 (%db)</td>
<td>NA (%wb)</td>
<td>7.25 (%db)</td>
<td>6.2 (%wb)</td>
<td>6.5 (%wb)</td>
<td>9.73 (%wb)</td>
</tr>
<tr>
<td>Crude protein</td>
<td>16.8 (19)</td>
<td>19 (%db)</td>
<td>1.13 (%wb)</td>
<td>16.8 (19)</td>
<td>1.3 (19)</td>
<td>23.5 (19)</td>
<td>1.23 (19)</td>
</tr>
<tr>
<td>Carbohydrate</td>
<td>NA</td>
<td>65 (%db)</td>
<td>2.26 (%wb)</td>
<td>NA (19)</td>
<td>2.8 (19)</td>
<td>NA (19)</td>
<td>NA (19)</td>
</tr>
<tr>
<td>Fat</td>
<td>8.1 (19)</td>
<td>9.2 (19)</td>
<td>1.09 (19)</td>
<td>NA (19)</td>
<td>1.3 (19)</td>
<td>16.7 (19)</td>
<td>1.8 (19)</td>
</tr>
<tr>
<td>Ash</td>
<td>1.0 (19)</td>
<td>6.7 (19)</td>
<td>NA (19)</td>
<td>11.4 (%db)</td>
<td>0.8 (19)</td>
<td>10.5 (19)</td>
<td>0.9 (19)</td>
</tr>
<tr>
<td>NDF</td>
<td>11.7 (19)</td>
<td>13.3 (19)</td>
<td>NA (19)</td>
<td>NA (19)</td>
<td>NA (19)</td>
<td>NA (19)</td>
<td>1.2 (19)</td>
</tr>
<tr>
<td>Starch</td>
<td>22.0 (19)</td>
<td>25.1 (%db)</td>
<td>NA (19)</td>
<td>NA (19)</td>
<td>NA (19)</td>
<td>NA (19)</td>
<td>NA (19)</td>
</tr>
</tbody>
</table>

Rausch et al (2013) found that starch had a higher fouling rate compared to sucrose. The results were supported by further experimentation where carbohydrate was added to commercial thin stillage. Starch had a higher effect on thin stillage fouling than sucrose. Challa et al (2015) investigated effects of carbohydrate mixtures (starch, glucose and corn syrup solids) on fouling. Starch was found to be the major constituent that caused fouling. Waxy starches were found to have longer induction periods and higher maximum fouling resistances than high amylose starches. Use of model fluids to study the effect of single or multiple compositions on fouling also was used in fouling studies of dairy and oil refinering industries (Bansal and Chen 2006a; Deshannavar et al 2010).

2.7 Fouling in the oil refinery industry

Heat exchanger fouling is a major concern for petroleum refineries. Fouling in crude oil
refining was estimated to cost $3.6 billion/yr in 2014 with more than 60% from oil preheat trains (PHTs), where petroleum is separated by distillation (Coletti and Hewitt 2015). Chemical reaction fouling is the dominant deposition mechanism in these heat exchangers (Wang et al 2015).

Most fouling experiments on crude oil fouling were established by laboratory experimental units. These fouling tests were designed to achieve accelerated fouling under controlled operating conditions. According to Deshannavar et al (2010), recycle flow loops with annular flow geometry had been used because they were better to visualize fouling deposits and collection of deposits was easier. Surface temperature, bulk velocity, bulk temperature and crude blending were reported to have the most influences on crude oil fouling.

Mathematical models were used to predict fouling rates of PHTs (Aminian and Shahhosseini 2008; Deshannavar et al 2010; Ishiyama et al 2010; Coletti and Hewitt 2015; Wang et al 2015). The concept of a “threshold” fouling model was introduced by Ebert and Panchal (1997). Two competing mechanisms controlling the incidence of fouling were included; chemical reactions promoted fouling while shear stresses at the tube surface tended to mitigate fouling. The threshold was defined as a stage when both mechanisms were balanced, so the net rate of fouling was zero. If the situation in a heat exchanger was above the threshold, fouling deposits increased (Ebert and Panchal 1997). The threshold fouling model allowed estimation of operating conditions where the fouling rate would be close to zero, termed the “fouling threshold”. This information would offer a potential rationale and quantitative basis to improve unit operation of heat exchangers (Wilson et al 2005). They reported the basic threshold model had been revised and had many variations to allow designers and operators to use quantitative criteria to select appropriate operating conditions and mitigate fouling. Limitations of these
models in design are the variability and uncertainty in the models as well as the lack of reliable data on tube and shell side pressure drops.

2.8. Fouling in the dairy industry

Fouling in dairy industry is so intense that heat exchangers need to be cleaned daily (Bansal and Chen 2006a). As a result, additional costs due to interruptions dominate dairy fouling costs (Georgiadis et al 1998).

Two types of milk fouling take place: protein (type A) and mineral (type B). Type A deposits formed between 75 and 105°C are a white precipitate composed of 60 to 80% protein, 15 to 35% minerals and 4 to 8% fat. At higher temperatures (> 110°C), mineral fouling takes place. Those deposits, which are composed of 70 to 80% mineral (mainly calcium phosphate), 15 to 20% protein and 4 to 8% fat, have a hard granular structure and are gray in color (de Jong 1997; Visser and Jeurnink 1997; Bansal and Chen 2006a; Sadeghinezhad et al 2015).

Model fluids of fixed compositions were used (Dausin et al 1987; Dalgleish 1990; Belmar-Beiny et al 1993; Bansal and Chen 2006b) to mimic milk fouling due to the natural variations of milks. β-lactoglobulin (β-Lg), whey protein concentrates (WPC), skim milk powder. Belmar-Beiny et al (1993) suggested protein aggregation was the governing reaction that controlled fouling. According to Bansal and Chen (2006a), fouling was considered to depend on protein reactions as well as mass transfer. Heat sensitive whey proteins were major components in dairy fouling deposits (~55% of deposits) (Visser and Jeurnink 1997) and were found to affect fouling (Sadeghinezhad et al 2015). Gotham et al (1992) found that β-Lg was the dominant protein that affected fouling because of its high heat sensitivity which lead to denaturation and aggregation. Bansal and Chen (2006) concluded that calcium phosphate played important roles
by interacting with β-Lg, thus enhancing the formation of deposits. Sadeghinezhad et al (2015) concluded that deposition proceeded through a complex process in which both whey protein aggregation and calcium phosphate formation were relevant.

Air bubbles formed on the heat transfer surface enhance fouling. (Bansal and Chen 2006a; Sadeghinezhad et al 2015) Fouling decreased with increasing turbulence as higher flow rates promoted deposit detachment through enhanced fluid shear stress. Bansal and Chen (2006b) found absolute temperature and temperature differences were crucial to fouling. Surface temperature was the most important factor in initiating fouling.

2.8.1. Effects of temperature in milk fouling

Belmar-Beiny et al (1993), using whey protein concentrates, discovered that total amount of fouling deposits increased with inlet temperatures. Chen and Bala (1998) investigated effects of surface and bulk temperatures on fouling of whole milk, skimmed milk and whey protein solution. Different combinations of surface and bulk temperatures were applied. When the bulk temperature was higher than the surface temperature, surface temperature was important to the weight of deposit. When the surface temperature was higher than bulk temperature, dry deposit weight decreased with the increase of bulk temperature. Fouling deposits began to be noticeable when surface temperature was above 65°C. Surface temperature played a more important role than bulk temperature as to the initiation of a fouling process.
CHAPTER 3. SURFACE AND BULK TEMPERATURE EFFECTS ON FOULING CHARACTERISTICS OF THIN STILLAGE

3.1. Introduction and objectives

3.1.1 Introduction

Fouling of heat transfer surfaces is a complex phenomenon that decreases heat transfer efficiency and increases capital and maintenance costs (Epstein 1983), decreases production capacity or even quality. Fouling was estimated to cost $14 billion in the US annually (Awad 2011). Although heat transfer fouling has been studied extensively since the 1900s (Somerscales 1990), there are few studies that were focused on fouling in the corn processing industry. In the US, there are more than 200 corn ethanol processing plants (RFA 2015) and evaporator fouling was reported to be a chronic problem as it decreased evaporator efficiency, increased steam consumption and periodic cleaning is needed (Singh et al 1999; Agbisit et al 2003; Wilkins et al 2006a; Wilkins et al 2006b). Previous studies on thin stillage fouling have been focused on effects of thin stillage composition, pH and fluid flow properties. In dairy and petrochemical industries, temperature is an important factor affecting fouling (Bansal and Chen 2006a; Simmons et al 2007; Deshannavar et al 2010). An increase of bulk temperature in the milk enhanced rates of protein denaturation and aggregation.

An annular fouling probe was used to monitor fouling resistance, which is used in fouling research (Panchal and Watkinson 1993; Singh et al 1999; Wilkins et al 2006a; Wilkins et al 2006b; Arora et al 2010; Rausch et al 2013; Challa et al 2015). Because deposits formation in milk was found due to seasonal variations in milk composition and fouling characteristics varied
from day to day, model test fluids were used in dairy industry to eliminate those variations of milk and to uncover fouling mechanisms in dairy processing (Visser and Jeurnink 1997; Simmons et al 2007). Rausch et al (2013) and Challa et al (2015) used model thin stillage of carbohydrates mixtures and starch was found to have largest effects on fouling tendencies. Together with commercial thin stillage samples, 1% starch solution was used to study temperature effects. Based on knowledge from fouling in dairy, we hypothesized temperature conditions would affect thin stillage fouling characteristics. Specifically, higher bulk and initial probe temperatures will intensify fouling results with increased fouling rates and decreased induction periods.

3.1.2 Objectives

The objectives were to:

1. Examine effects of bulk and initial probe temperature on commercial and model thin stillage fouling characteristics.
2. Determine bulk and initial probe temperature needed for repeatable and rapid characterization of fouling behavior.

3.2. Methods and materials

3.2.1. Test apparatus

The system was used to detect fouling under accelerated conditions, which were achieved by recycling test fluid under temperature conditions (bulk temperature 60 to 80°C) similar to the operating temperature (75°C) typical to a dry grind facility. Initial surface temperature or probe
temperature were 100 and 120°C. The condition of surface temperatures are more severe than that in the multiple effect evaporators in the dry grind plant. Fouling test apparatus was similar to that used in previous research (Agrisit et al 2003; Wilkins et al 2006a; Wilkins et al 2006b; Arora et al 2010; Rausch et al 2013; Challa et al 2015). The system consisted of an annular probe, 20 L batch tank, centrifugal pump and heat exchanger (Fig. 3.1). Test fluid was circulated from a 20 L reservoir tank by a centrifugal pump (S-115 RZ, Iwaki Walchem, Iwaki, Japan). A water bath was used to heat the test fluid to desired temperatures and a cooling coil was used to obtain the heat balance within the flow loop and maintain bulk temperature. The annular fouling probe (FIREROD 1025, Watlow, St. Louis, MO) consisted of a stainless steel (SS 316) outer tube containing a resistance heater (208V, 2000W).

![Figure 3.1. Schematic of fouling test system (TC = thermocouple)](image)

Fluid flow through the rod in the annular space between the rod and outer housing tube. The inner rod contained an electrical resistance heater and five type K thermocouples embedded in the inner wall of the rod. Four of the thermocouples were used to measure inner wall
temperature at four locations on the probe surface. The fifth thermocouple was used to cut off power supply to the heater rod when probe temperature reached 200°C. Temperature readings from the thermocouple were recorded every min and were used to calculate the fouling resistance.

Fouling resistances were calculated using equations described in section 2.5.2. The x/k values which were used to calculate fouling resistance (Table 3.1) were determined using methods described in Fischer et al (1975).

Table 3.1. The x/k values of each thermocouple.

<table>
<thead>
<tr>
<th>Thermocouple number</th>
<th>x/k* (m²K/kW)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TC1</td>
<td>0.0749</td>
</tr>
<tr>
<td>TC2</td>
<td>0.1095</td>
</tr>
<tr>
<td>TC3</td>
<td>0.0971</td>
</tr>
<tr>
<td>TC4</td>
<td>0.0976</td>
</tr>
</tbody>
</table>

* x/k is the distance of the thermocouple from the surface divided by the thermal conductivity of the probe metal

3.2.2. Cleaning

After each fouling test, a plastic spatula was used to scrape fouling deposits from the probe surface. After most of the deposits was removed, the rod was soaked in 5% (w/v) NaOH solution at room temperature overnight. After soaking, all remaining deposits was removed using a wet sponge. After cleaning the probe, hot tap water (50 to 70°C) was circulated throughout the
system to check for leaks and drain the deposits out of the system. Detergent solution (10 L 1% w/v), (Alconox, New York, NY) was circulated for 20 min for further cleaning. Hot tap water (15 L) was circulated for 20 min followed by a 150 L hot tap water rinse. Hot tap water (50°C) flowed in and out of the system continuously during the rinse. Throughout cleaning, flow rate was adjusted to maximum capacity (4.5 to 5.0 gal/min) for better cleaning results. After cleaning all water was drained from the system.

3.2.3. Fouling of commercial thin stillage

3.2.3.1. Materials

Thin stillage samples were collected from a commercial dry grind facility. Thin stillage sample was stored at 4°C for a period of 1 to 2 wk in the previous studies (Singh et al 1999; Wilkins et al 2006a; Wilkins et al 2006b; Arora et al 2010). Zheng (2013) stored commercial thin stillage samples at room temperature and study effects of aging. No differences of fouling characteristics were observed for sample stored from 1 to 20 days. For this study thin stillage samples were stored at room temperature (15 ± 5°C) and tested within 7 days. Five batches (50 L) were collected separately during a 2 mo period with four tests conducted per batch (Table 3.3). A 10 L batch sample was used for each fouling test. Total solids were measured using a standard method (AACCI 2000). Solids measurements were repeated three times and mean values were used as the total solids contents.

3.2.3.2. Experiment procedure and treatment conditions

The fouling test of commercial thin stillage was started after the system was cleaned. Commercial thin stillage (10L) sample was added to the tank. The thin stillage sample was mixed by circulating at maximum flow rate (4 to 5 gal/min) for 5 min. Sample volume was
reduced to 7 L by draining. Water bath was turned on to heat the fluid to desired bulk temperatures. Density and viscosity were measured after fluid reaching the bulk temperature for each treatment. Study showed Reynolds number had effects on thin stillage fouling (Wilkins et al 2006a). Reynolds number was kept in a constant range (460 to 520) for each treatment to eliminate effects of Reynolds number. Tap water was introduced in the heat exchange system to maintain the bulk temperature.

Temperature conditions with randomized treatments are shown in Table 3.2. Bulk temperature ($T_b$) was adjusted to desired treatment conditions ($60 \pm 2^\circ C$ and $80 \pm 2^\circ C$) and was kept constant throughout the test periods. After reaching the desired bulk temperature, flow rate was adjusted and the probe power supply was turned on. Test was initiated when initial probe temperature ($T_i$) reached desired conditions ($100 \pm 2^\circ C$ and $120 \pm 2^\circ C$). Each test lasted for a period of 5 hr. The bulk temperature $80^\circ C$ and initial temperature $120^\circ C$ were close to the temperature conditions Challa (2015) used (bulk temperature $75^\circ C$, initial probe temperature $120^\circ C$) for fouling tests. Initial probe temperature was difficult to maintain when bulk temperature was lower than $60^\circ C$. The largest temperature difference between bulk temperature and initial probe temperature for the system to be stable was $60^\circ C$. During each test fouling, data (bulk temperature, wall temperature and power input) were recorded every min with the data logger.

After each experiment, the fouling probe was removed from the outer tube and fouling deposits were removed using a plastic spatula. The probe was soaked in 5% (w/v) NaOH solution overnight after removing most deposits. Remaining deposits were removed using a wet sponge after soaking. Cleaning procedure was described in section 3.2.2.
Table 3.2. Experimental design (randomized complete block) for thin stillage fouling tests.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Batch 1</th>
<th>Batch 2</th>
<th>Batch 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T_i^a = 120$, $T_b^a = 80$</td>
<td>1*</td>
<td>2</td>
<td>4</td>
</tr>
<tr>
<td>$T_i = 120$, $T_b = 60$</td>
<td>2</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>$T_i = 100$, $T_b = 80$</td>
<td>3</td>
<td>4</td>
<td>1</td>
</tr>
<tr>
<td>$T_i = 100$, $T_b = 60$</td>
<td>4</td>
<td>3</td>
<td>3</td>
</tr>
</tbody>
</table>

$^a T_i = $ initial probe temperature (°C) $T_b = $ fluid bulk temperature (°C)

* 1, 2, 3, 4 is the order of tests for each batch

3.2.4. Fouling of model thin stillage

3.2.4.1 Materials

Model thin stillage was used in previous work (Rausch et al 2013; Challa et al 2015) as a repeatable experiment material. Model thin stillage of commercial corn starch had rapid fouling compared with other carbohydrate mixtures. Regular yellow dent maize starch (obtained from Tate & Lyle, Decatur, IL, US) slurry (1% dry basis) was used as model thin stillage to study effects of bulk and surface temperatures on fouling. Starch solution was prepared at the beginning of each test.

3.2.4.2. Experimental procedure and treatment conditions

After cleaning, 7 L tap water was circulated and preheated to desired bulk temperatures ($T_b = 60$ or 80°C) in the system. Starch (70 g) was added slowly into the tank to form 1% db starch slurry. The slurry was circulated by the pump at maximum flow rate (4 gal/min) for 30 min. Experiment started when the probe power was turned on and reached the desired initial probe temperature ($T_i = 100$ or 120°C).
Treatments were arranged in a randomized complete block design with three replications for each treatment (Table 3.3). Viscosity and density were tested each time before recording data. Reynolds number (480 ± 20) was kept constant for each test. Each test ended after 5 hr or when inner wall temperature reach 200°C. Using data logger, bulk temperature, wall temperature and power input were recorded every min, which were used to calculate fouling resistance (see section 2.5.2). After each test, same cleaning procedure was conducted with experiments for commercial thin stillage.

Table 3.3. Experimental design (randomized complete block) for model thin stillage fouling test.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Replication 1</th>
<th>Replication 2</th>
<th>Replication 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>T&lt;sub&gt;i&lt;/sub&gt;=120, T&lt;sub&gt;b&lt;/sub&gt;=80</td>
<td>1*</td>
<td>2</td>
<td>4</td>
</tr>
<tr>
<td>T&lt;sub&gt;i&lt;/sub&gt;=120, T&lt;sub&gt;b&lt;/sub&gt;=60</td>
<td>2</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
<td>T&lt;sub&gt;i&lt;/sub&gt;=100, T&lt;sub&gt;b&lt;/sub&gt;=80</td>
<td>3</td>
<td>4</td>
<td>1</td>
</tr>
<tr>
<td>T&lt;sub&gt;i&lt;/sub&gt;=100, T&lt;sub&gt;b&lt;/sub&gt;=60</td>
<td>4</td>
<td>3</td>
<td>2</td>
</tr>
</tbody>
</table>

<sup>a</sup> T<sub>i</sub> = initial probe temperature (°C) T<sub>b</sub> = fluid bulk temperature (°C)

*1, 2, 3, 4 is the order of tests for each batch

3.2.5. Analysis methods

Statistical analyses were performed using statistical software (RStudio 0.99.447, RStudio, Boston, MA) with a significance level of p<0.05. Fouling resistance (R<sub>f</sub>) vs time was plotted for each test. Fouling rate was determined as the slope of the linear regression line from time 0 to time t. Mean fouling resistance was calculated as an average of three replicates for each data point and mean fouling resistances vs time were plotted (Fig. 3.2) to demonstrate the overall tendencies of fouling under different temperature treatments. To illustrate fouling characteristics,
fouling rates were calculated with two methods, a linear regression line with an intercept of 0 (setting the intercept as “0” while fitting regression line in Microsoft Excel 2013) and without a 0 intercept which was used by Wilkins (2005).

Figure 3.2. Example of calculating fouling resistance

Linear fouling rates for 1, 2 and 5 hr were calculated, defined as FR1, FR2 and FR5, respectively. Induction period was defined as the period of time during which the 3 min moving average of $R_f$ was less than 0.05 m$^2$·K/kW (Challa et al 2015). Wilkins (2005) defined induction period to be the 1 min moving average of $R_f$ was less than 0.01 m$^2$·K/kW. Maximum fouling resistance ($R_{\text{max}}$) was defined as the largest value of the 3 min moving average of fouling resistance during the 5 hr test period which also was used by Challa (2015). Sloughing point (SP) was defined as the point of time where fouling resistance decreased by more than 30%.
One representative of fouling curve of commercial thin stillage fouling at higher temperatures ($T_i = 120$, $T_b = 80^\circ C$) was shown in Fig. 3.2. As a result of deposit sloughing, when calculating the fouling rate for 5 hr, without setting the intercept as 0, the slope of the linear regression line was negative (Fig. 3.2). For this reason we use fouling rate in 1, 2, 5 hr with intercept of 0 to better illustrate fouling behavior.

![Fouling curve and analysis](image)

**Figure 3.3.** Example of fouling curve and analysis (one representative of model thin stillage sample tested at $T_i=120$, $T_b=80^\circ C$)

One representative of fouling curve of model thin stillage fouling at high temperatures ($T_i = 120$, $T_b = 80^\circ C$) was shown in Fig 3.3. Fouling resistance decreased after reaching maximum fouling resistance and gradually decreased throughout rest of testing periods. Negative fouling rates were calculated because of the decrease of fouling resistance when we do not set the intercept as 0.
intercept. For better analyses, all fouling rates (FR1, FR2 and FR5) were calculated with a y-axis intercept of 0.

Fouling rates, maximum fouling resistance and induction periods were calculated for each treatment. ANOVA was calculated for fouling rates, induction periods and max fouling resistance as dependent variables; bulk temperature and initial probe temperature as independent variables, to determine the effect of bulk temperature and initial probe temperature on fouling characteristics independently. Fisher’s least significant method was used to determine if induction periods, maximum fouling resistance of different bulk and initial probe temperature were different. Means of maximum fouling resistance, fouling rates and induction periods of different treatments were compared using one-way ANOVA and TukeyHSD tests.

3.3. Results and discussion

3.3.1. Temperature effects on commercial thin stillage fouling

When $T_i = 120^\circ$C and $T_b = 80^\circ$C, fouling resistance increase rapidly during the first 2 hr period (Fig. 3.4). The rapid increase of fouling resistance could be quantified using the 1 and 2 hr fouling rates (FR1 = 0.38 m$^2$·K/kW·hr, FR2 = 0.25 m$^2$·K/kW·hr, Table 3.5). Fouling resistance ceased to increase after reaching a maximum fouling resistance of 0.47 m$^2$·K/kW. A sudden decrease of more than 60% of fouling resistance could be due to sloughing of thin stillage components which also was observed by (Challa et al 2017).

When $T_i = 120^\circ$C and $T_b = 60^\circ$C, fouling resistance increased less rapidly as in the condition of $T_i = 120^\circ$C and $T_b = 80^\circ$C (Fig. 3.4). This can be quantified by fouling rates in 1 and 5 hr (FR1 = 0.023, FR2 = 0.022 m$^2$·K/kW·hr, Table 3.4), which were lower than FR1 and FR2 at
higher $T_b$ (80°C), respectively. Induction periods of 1.8 hr were observed. $R_{\text{max}}$ (0.36 m$^2$·K/kW) was reached at final stages during 5 hr fouling tests. Deposit sloughing occur for one replicates at the end of test period ($t = 4.5$ hr).

Figure 3.4. Fouling resistance for commercial thin stillage over time (TS: thin stillage; a,b,c: three replicates from three batches (1,2,3); eg, TS120-80a means thin stillage sample from batch 1 tested at initial probe temperature 120°C and bulk temperature 80°C).

At $T_i = 100^\circ$C, fouling rates (FR1, FR2 and FR5) and $R_{\text{max}}$ were lower than those at 120°C (Table 3.4). Induction periods were more than 5 hr for tests at $T_b = 60^\circ$C.

Variations were observed (CV shown in Table 3.4) for each batch of thin stillage,
attributed to variations in composition of thin stillage from batch to batch. Mean fouling curves for each temperature treatment had an increase of fouling (characterized by fouling resistance) with increase of \( T_i \) (from 60 to 80°C). An increase of fouling rate at higher \( T_b \) (80°C) was observed when the \( T_i = 120°C \).

Table 3.4. Mean fouling rates, induction periods and maximum fouling resistance of commercial thin stillage.

<table>
<thead>
<tr>
<th>( T/T_b ) (^{1}) (°C)</th>
<th>( FR1^2 ) (m(^2)-K/kW·hr)</th>
<th>CV (^3) of ( FR1 ) (%)</th>
<th>( FR2^2 ) (m(^2)-K/kW·hr)</th>
<th>CV of ( FR2 )</th>
<th>( FR5^2 ) (m(^2)-K/kW·hr)</th>
<th>CV of ( FR5 )</th>
<th>( R_{\text{max}}^4 ) (m(^2)-K/kW)</th>
<th>CV of ( R_{\text{max}} )</th>
<th>IP (^5) (hr)</th>
<th>CV of IP</th>
</tr>
</thead>
<tbody>
<tr>
<td>120/80</td>
<td>0.380a*</td>
<td>15.0</td>
<td>0.2500a</td>
<td>16</td>
<td>0.0720a</td>
<td>35</td>
<td>0.470a</td>
<td>0.081</td>
<td>0.14a</td>
<td>110.00</td>
</tr>
<tr>
<td>120/60</td>
<td>0.0230b</td>
<td>5.0</td>
<td>0.0220b</td>
<td>45</td>
<td>0.0470a</td>
<td>37</td>
<td>0.360a</td>
<td>0.400</td>
<td>1.80b</td>
<td>30.00</td>
</tr>
<tr>
<td>100/80</td>
<td>0.0210b</td>
<td>150.00</td>
<td>0.0150b</td>
<td>130.0</td>
<td>0.0090b</td>
<td>130.0</td>
<td>0.045b</td>
<td>1.100</td>
<td>3.80c</td>
<td>54.00</td>
</tr>
<tr>
<td>100/60</td>
<td>0.0016b</td>
<td>240.00</td>
<td>0.0018b</td>
<td>73</td>
<td>0.0041b</td>
<td>530.0</td>
<td>0.028b</td>
<td>0.670</td>
<td>5.00c(^6)</td>
<td>--</td>
</tr>
</tbody>
</table>

\(^{1}\)T\(_i\) = initial probe temperature, \( T_b \) = bulk temperature  
\(^{2}\)FR1, FR2 and RF5 = fouling rate in 1, 2 and 5 hr (set intercept)  
\(^{3}\)CV = coefficients of variation  
\(^{4}\)\( R_{\text{max}} \) = maximum fouling resistance  
\(^{5}\)IP = induction period  
\(^{6}\)5.00 = longer than 5 hr  
*Means of three replicates, value with the same letter in the same column were not different (p<0.05)

\( T_i \) was a factor affecting fouling characteristics in the aspect of fouling rates, maximum fouling resistance and induction periods. (Fig. 3.5, 3.6, Appendix 3.1). \( T_b \) was a factor affecting fouling rate in 1 and 2 hr, and induction period (Fig. 3.5, 3.6, Appendix 3.1). When initial probe temperature was 120°C, higher bulk temperature (80°C) would increase initial fouling rates (FR1, FR2, Fig. 3.4). At lower \( T_b \), induction periods were longer (Table 3.4, Fig. 3.6b). Differences among \( R_{\text{max}} \) was not detected among treatments of different bulk temperature conditions. There were no effects of bulk temperature on maximum fouling resistance detected (Fig. 3.6a).
Figure 3.5 Mean fouling rates of 1 (a), 2 (b) and 5hr (c) in different temperature conditions for commercial thin stillage. Means of three replicates, value with the same letter in the same column were not different (p≤0.05)
Figure 3.6 Mean Maximum fouling resistance (a) and inductions period (b) in different temperature conditions for commercial thin stillage. Means of three replicates, value with the same letter in the same column were not different (p≤0.05)

Compared with previous studies, at $T_1 = 120^\circ$C, higher fouling rates and maximum fouling resistances were observed. Induction periods had a variation among the replicates (CV $\leq 110\%$, Table 3.4). Wilkins et al (2006a) and Arora et al (2010) also observed induction periods in their tests, while Challa (2014) did not observe induction periods (Table 3.5). The variation of induction period could be related with the initial conditions of the probe surface.
Table 3.5. Comparisons of fouling of thin stillage in temperature conditions, fouling rates and induction periods.

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Batch size (L)</td>
<td>30</td>
<td>30</td>
<td>30</td>
<td>50</td>
<td>7</td>
</tr>
<tr>
<td>Total solids (%)</td>
<td>NA</td>
<td>8.54 ± 0.18</td>
<td>7.30 ± 0.1</td>
<td>7.00</td>
<td>7.33 ± 0.16</td>
</tr>
<tr>
<td>Re /flow rate</td>
<td>0.26 m/sec</td>
<td>440</td>
<td>NA</td>
<td>1600</td>
<td>360 ± 10</td>
</tr>
<tr>
<td>T_b (ºC)</td>
<td>40 ± 2.5</td>
<td>40 ± 2</td>
<td>60 ± 2</td>
<td>52 ± 2</td>
<td>75 ± 2</td>
</tr>
<tr>
<td>T_i (ºC)</td>
<td>NA</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>120 ± 2</td>
</tr>
<tr>
<td>Test period (hr)</td>
<td>12.83</td>
<td>≤ 5</td>
<td>10</td>
<td>10</td>
<td>5</td>
</tr>
<tr>
<td>Induction period (hr)</td>
<td>NA</td>
<td>0.041 to 0.70</td>
<td>0.73 ± 0.09</td>
<td>NA</td>
<td>No</td>
</tr>
<tr>
<td>R_max (m²·K/kW)</td>
<td>0.35</td>
<td>0.4</td>
<td>0.37 ± 0.010 (m²·C/kW)</td>
<td>0.25 to 0.30</td>
<td>*0.16</td>
</tr>
<tr>
<td>FR (m²·K/kWh)</td>
<td>0.021</td>
<td>0.091 ± 0.024</td>
<td>0.043 (m²·C/kWh)</td>
<td>NA</td>
<td>0.024</td>
</tr>
</tbody>
</table>

T_b = bulk temperature of the test fluid.
T_i = initial probe temperature.
R_max = maximum fouling resistance.
* Not a value presented by the author; estimated from the graphs in publication.

3.3.2. Temperature effects on model thin stillage fouling

At T_i = 120ºC and T_b = 80ºC, fouling deposits accumulated rapidly indicated by the rapid increase of fouling resistance (FR1=0.91 m²·K/kW·hr). Induction periods were less than 5 min (Table 3.6). Fouling resistance decreased after reaching maximum resistance 0.71 m²·K/kW indicating deposit removal from the probe surface (Challa et al 2015) or the rate of removal was faster than the rate of deposition.
Figure 3.7. Temperature effects on model thin stillage (1% starch) (eg, M100-80a means model thin stillage sample tested at initial probe temperature 100°C and bulk temperature 80°C as replicate 1)

At $T_i = 120^\circ$C and $T_b = 60^\circ$C, there were induction periods of about 1 hr (Table 3.6).

Fouling rate decreased near to 0 after reaching maximum fouling resistance 0.36 m$^2$·K/kW. Fouling resistance remained the same for the rest of test periods (Fig. 3.7).

When initial probe temperature was 100°C, induction periods were longer than 5 hr for all tests. Maximum fouling resistance was lower than 0.05 m$^2$·K/kW. No observable deposits were found on the probe surface after the 5 hr tests.
Table 3.6. Mean fouling rates and maximum fouling resistances of model thin stillage in different temperature conditions.

<table>
<thead>
<tr>
<th>Temperature</th>
<th>(^1\text{T}/\text{T}_b)</th>
<th>(^2\text{FR1})</th>
<th>(^3\text{CV})</th>
<th>(^2\text{FR2})</th>
<th>(^3\text{CV}) of (^2\text{FR2})</th>
<th>(^2\text{FR5})</th>
<th>(^3\text{CV}) of (^2\text{FR5})</th>
<th>(^4\text{R}_{\text{max}})</th>
<th>(^3\text{CV}) of (^4\text{R}_{\text{max}})</th>
<th>(^5\text{IP})</th>
<th>(^3\text{CV}) of (^5\text{IP})</th>
</tr>
</thead>
<tbody>
<tr>
<td>120/80</td>
<td>0.91a*</td>
<td>0.11</td>
<td>0.42a</td>
<td>0.17</td>
<td>0.13a</td>
<td>0.12</td>
<td>0.71a</td>
<td>0.10</td>
<td>0.083a</td>
<td>0.40</td>
<td></td>
</tr>
<tr>
<td>120/60</td>
<td>0.0087b</td>
<td>3.6</td>
<td>0.18b</td>
<td>0.12</td>
<td>0.068a</td>
<td>0.67</td>
<td>0.36b</td>
<td>0.082</td>
<td>1.03b</td>
<td>0.22</td>
<td></td>
</tr>
<tr>
<td>100/80</td>
<td>0.012b</td>
<td>0.78</td>
<td>0.0077c</td>
<td>0.85</td>
<td>0.00043b</td>
<td>8.3</td>
<td>0.020c</td>
<td>0.56</td>
<td>5c</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>100/60</td>
<td>0.0089b</td>
<td>0.70</td>
<td>0.0044c</td>
<td>0.30</td>
<td>0.0043b</td>
<td>0.29</td>
<td>0.023c</td>
<td>0.31</td>
<td>5c</td>
<td>0</td>
<td></td>
</tr>
</tbody>
</table>

\(^1\text{T}_i=\text{initial probe temperature, } \text{T}_b=\text{bulk temperature}\)
\(^2\text{FR1, FR2 and RF5= fouling rate in 1, 2 and 5 hr (set intercept)}\)
\(^3\text{CV}=\text{coefficients of variation}\)
\(^4\text{R}_{\text{max}}=\text{maximum fouling resistance}\)
\(^5\text{IP}=\text{induction period}\)
\(^*\text{Means of three replicates, value with the same letter in the same column were not different (p≤0.05)}\)

\(\text{T}_i\) was a factor in fouling characteristics in the aspect of fouling rates (1, 2 and 5 hr), maximum fouling resistance and induction periods. \(\text{T}_b\) was significant in fouling rate of 1 and 2 hr but not in 5 hr fouling rate. This corresponded with previous analysis that fouling rates and maximum fouling resistances increased with the increase of initial probe temperature, while induction periods decreased. Decrease of bulk temperature would result in a decrease of maximum fouling resistance and increased induction periods (Fig 3.9).
Figure 3.8. Mean fouling rates of 1 (a), 2(b) and 5hr (c) in different temperature conditions for model thin stillage. Means of three replicates, value with the same letter in the same column were not different (p≤0.05)

Maximum fouling resistance of 0.71 m$^2$·K/kW was higher than that reported by Challa et al. (2015) (0.41 m$^2$·K/kW) with the same $T_i$ (120°C) and slightly lower $T_b$ (75°C). Longer induction periods (300 min) were observed for tests under $T_i=120$ and $T_b=80$, than previous studies which were less than 5 min (Table 3.7).
Figure 3.9 Mean Maximum fouling resistance (a) and inductions period (b) in different temperature conditions for model thin stillage. Means of three replicates, value with the same letter in the same column were not different (p≤0.05).

Table 3.7. Comparison of fouling parameters with previous studies using model thin stillage.

<table>
<thead>
<tr>
<th>Test</th>
<th>( ^2 T_i (\degree C) )</th>
<th>( ^2 T_b (\degree C) )</th>
<th>( ^3 FR )</th>
<th>( ^4 R_{\text{max}} )</th>
<th>( ^5 \text{IP} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Present study</td>
<td>1% starch</td>
<td>120 ± 2</td>
<td>80 ± 2</td>
<td>0.13</td>
<td>0.71</td>
</tr>
<tr>
<td>Present study</td>
<td>1% starch</td>
<td>120 ± 2</td>
<td>60 ± 2</td>
<td>0.068</td>
<td>0.36</td>
</tr>
<tr>
<td>Present study</td>
<td>1% starch</td>
<td>100 ± 2</td>
<td>80 ± 2</td>
<td>0.00043</td>
<td>0.020</td>
</tr>
<tr>
<td>Present study</td>
<td>1% starch</td>
<td>100 ± 2</td>
<td>60 ± 2</td>
<td>0.0043</td>
<td>0.023</td>
</tr>
<tr>
<td>Challa et al</td>
<td>1% starch</td>
<td>120 ± 2</td>
<td>75 ± 2</td>
<td>0.54</td>
<td>0.41</td>
</tr>
<tr>
<td>Rausch et al</td>
<td>1% starch</td>
<td>100 ± 2</td>
<td>50 ± 2</td>
<td>NA</td>
<td>0.25-0.3</td>
</tr>
</tbody>
</table>

1previous study, all study was using 1% starch solution as model thin stillage test fluid and using an annular fouling test apparatus

2\( T_i \) = initial probe temperature, \( T_b \)=bulk temperature

3\( FR \) = fouling rate, calculated differently (see method)

4\( R_{\text{max}} \) = maximum fouling resistance

5\( \text{IP} \) = induction period

Starch gelatinization begins above temperature of 60\( \degree \)C. Higher maximum fouling
resistance at bulk temperature may be due to starch gelatinization. Negative fouling resistance was observed at the beginning of tests when initial probe temperature was 120ºC and bulk temperature was 80ºC. This was observed when temperature difference of bulk temperature and initial probe temperature was the largest (60ºC). Whether or not the negative fouling resistance was caused by increased temperature difference at the beginning of tests was not clear. Negative $R_f$ value was reported in other’s work (Singh et al 1999; Wilkins et al 2006a; Wilkins et al 2006b; Arora et al 2010; Rausch et al 2013). Negative fouling rates were explained by Crittenden and Alderman (1987) as a phenomenon caused by the enhancement of heat transfer due to increase of roughness of heat transfer surface in early stage of fouling.

3.4. Conclusions

$T_i$ and $T_b$ had effects on commercial and model thin stillage fouling characteristics. Fouling rates and maximum fouling resistance increased with the increase of initial probe temperature for commercial and model thin stillage. For model thin stillage, increasing bulk temperature would increase maximum fouling resistance. Bulk temperatures did not affect maximum fouling resistance for commercial thin stillage. Lower bulk temperatures increased induction periods in commercial and model thin stillage fouling.

At $T_i = 120^\circ$C and $T_b = 80^\circ$C, rapid fouling was observed for commercial and model thin stillage samples. At $T_i = 100^\circ$C and $T_b = 80$ and $60^\circ$C, little or no fouling occurred for both test samples.

For commercial thin stillage, $T_i = 120^\circ$C and $T_b = 80^\circ$C would be the recommended temperature conditions because of rapid fouling and less variation in fouling parameters. For
future fouling tests of model thin stillage (1% starch), T_i = 120°C and T_b = 60°C would be the recommended temperature conditions because of the rapid fouling and repeatable induction periods.
CHAPTER 4. EVAPORATOR HEAT TREATMENT EFFECTS ON FOULING CHARACTERISTICS OF THIN STILLAGE

4.1. Introduction and objectives

Challa (2014) examined fouling of commercial thin stillage and concentrate from individual effects of multiple effect evaporator. Samples were collected from different locations of a multiple effect evaporator in a dry grind plant. Each sample was tested with different total solids (from 7 to 11%). Higher fouling rates were observed in samples with higher total solids.

Heat treatment influence protein aggregation and were found to affect deposition of solid materials in milk processing (Burton 1968; Lucey et al 1999). Time exposed to heat surfaces were found to be a factor affecting fouling in general (Kukulka and Devgun 2007).

Challa (2014) observed a change of fouling characteristics before and after plant shut down and cleaning. Fouling rates of thin stillage and concentrates after plant shut down were similar and induction periods were more than 5 hr. He concluded that evaporator cleaning decreased fouling rates of thin stillage and concentrates but it was unclear if plant shut down had an effect on fouling behavior.

Our hypothesis was that heat treatment of the evaporator would affect fouling characteristics. We also hypothesized the complete shutdown and cleaning of the plant would reduce the rate of foulant accumulation once the plant restarted.

The objectives were to:

1. Examine fouling characteristics of commercial thin stillage and concentrates with
uniform solids content.

2. Observe effects of plant shut down and cleaning on fouling characteristics of samples from the thin stillage evaporator.

4.2. Materials and methods

4.2.1. Sample collection

Samples from different locations of a multiple effect evaporator were collected and diluted to the same solids content (7% ± 0.5) to eliminate the influence of solids content. Samples were collected from different locations of a multiple effect evaporator from a dry grind facility during a period of 90 days. Sample collection dates are listed in Table 4.1. The plant was shut down completely for 25 days and was cleaned (not the routine cleaning of evaporator every wk). In the facility, evaporation system had two effects. Each effect contained four stages (Fig 5.1); eight stages were connected in series. An oil skimming process took place between stages 7 and 8.

Table 4.1 Sample collection and plant cleaning date.

<table>
<thead>
<tr>
<th>Samples/ Plant operation</th>
<th>Date (2015)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Batch 1</td>
<td>10.01</td>
</tr>
<tr>
<td>Batch 2</td>
<td>10.08</td>
</tr>
<tr>
<td>Plant shut down and cleaning</td>
<td>10.11 to 11.15</td>
</tr>
<tr>
<td>Batch 3</td>
<td>11.18</td>
</tr>
<tr>
<td>Batch 4</td>
<td>12.02</td>
</tr>
<tr>
<td>Batch 5</td>
<td>12.14</td>
</tr>
</tbody>
</table>
Figure 4.1. Multiple effect evaporator in a dry grind facility. (a) First effect, (b) Second effect. (Adapted from Challa, 2017)
Samples (10 L) were collected at four locations for each batch (Fig. 4.1): thin stillage (TS) prior to the evaporators, concentrates from the end of evaporator effect 1 after stage 4 (E1), concentrates after skimming before entering stage 8 (SK) and concentrates from the end of evaporator effect 2 after stage 8 (E2), also known as condensed distillers solubles or syrup. Total solids contents were determined using a standard oven method (AACC 2000). Samples (20 ml) were dried in a 49°C oven overnight (12 hr) and further dried in a 135°C oven for 2 hr. Three determinations were made. Before each fouling test, samples were diluted with tap water to 7 ± 0.5% solids content, similar to thin stillage sample collected at the same batch. Samples were stored at room temperature (15 ± 5°C) and were tested within 7 days.

4.2.2. Fouling tests

To measure fouling resistance the annular fouling probe consisted of a stainless steel rod within a cylindrical housing. Sample fluid flow through the annulus cross section between outer wall and inner rod. An electrical resistance heater was positioned in the center of the rod. Thermocouples embedded in the rod were used to acquire temperature data which were recorded by a data logger. The test loop consisted of the annular probe, batch tank, heat exchanger, flow meter and centrifugal pump. Fouling test apparatus were identical to Chapter 3. Viscosity and density of the sample after diluting were tested when bulk temperature reached 75°C. Flow rates were adjusted (3 to 4 gal/min) to maintain a Reynolds number of 450 ± 50. Fouling tests were started when bulk temperature reached 80°C and initial probe temperature reached 120°C. Fouling tests lasted for 5 hr or when probe temperature reached 200°C. Bulk temperature was maintained constant during the tests (80 ± 2°C). Using a data logger, bulk temperature, probe surface temperature, power input were recorded every 1 min.
4.2.3. Data analysis

Treatments were arranged in a randomized complete block design (Table 4.2). Fouling resistance from each test was calculated using equations described in section 2.5.2. The slope of the linear regression line crossing point (0, 0) from time 0 to time t was defined as fouling rate (FR). This was accomplished by regression analysis in Microsoft Excel 2013 with the intercept set as 0. Fouling rate in 1 hr (FR1), 2 hr (FR2) and 5 hr (FR5) as well as R2 values of the regression line were calculated. Detailed method of R2 calculation can be found in appendix (A.3.1). An example of fouling rate calculation is shown in Fig. 4.2.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Batch 1</th>
<th>Batch 2</th>
<th>Batch 3</th>
<th>Batch 4</th>
<th>Batch 5</th>
</tr>
</thead>
<tbody>
<tr>
<td>TS1</td>
<td>1*</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>E12</td>
<td>2</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>2</td>
</tr>
<tr>
<td>SK3</td>
<td>3</td>
<td>4</td>
<td>4</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
<td>E24</td>
<td>4</td>
<td>3</td>
<td>1</td>
<td>2</td>
<td>1</td>
</tr>
</tbody>
</table>

TS1 = thin stillage,
E12 = concentrates at end of effect 1, after stage 4 of evaporator,
SK3 = concentrates after oil skimming before enter into stage 8,
E24 = concentrates at end of effect 2, after stage 8 of evaporator,
1*, 2, 3, 4 is the order of tests for each batch.

Sloughing point (SP) was defined as the point of time where fouling resistance decreased by more than 30%. First sloughing point (FSP) was the point of time where first sloughing took place. Fouling rate before deposit sloughing takes place (FRS) was defined as the slope of the linear regression line crossing (0, 0) from time 0 to the first sloughing point. Examples of fouling rate calculations and sloughing points are shown in Fig. 4.2. Maximum fouling resistance (Rmax)
was defined as the largest value of the 3 min moving average of fouling resistance during the 5 hr test periods. Induction period was defined as the period of time during which the 3 min moving average of $R_f$ was less than $0.05 \text{ m}^2\cdot\text{K/kW}$ (Challa et al 2015). Maximum fouling resistance and induction periods were calculated for each test. Means of fouling rates, maximum fouling resistances and induction periods before and after cleaning were calculated. One-way and two-way ANOVA were used to compare means. Statistical analyses were performed using RStudio (0.99.447, RStudio, Boston, MA) with a significance level of $p \leq 0.05$.

Figure 4.2. An example of fouling curve and analysis (FR1, FR2, FR5 = fouling rate in 1, 2, 5 hr. FRS = fouling rate before first sloughing point)
4.3. Results and discussion

Total solids contents of thin stillage and concentrates were stable and consistent throughout five batch samples with CV smaller than 6% (Table 4.3). The total solids content increased as thin stillage went through each stage of the evaporator. The solids content of thin stillage was 7.0% which is similar to Challa et al (2017). The solids contents of E2 was the highest (27.8%).

Table 4.3. Total solids contents of thin stillage and concentrates prior to dilution.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Batch 1</th>
<th>Batch 2</th>
<th>Batch 3</th>
<th>Batch 4</th>
<th>Batch 5</th>
<th>Mean</th>
<th>CV (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TS¹</td>
<td>6.70*a**</td>
<td>6.52a</td>
<td>7.49a</td>
<td>7.29a</td>
<td>7.35a</td>
<td>7.07</td>
<td>6.1</td>
</tr>
<tr>
<td>E1²</td>
<td>11.12b</td>
<td>11.01b</td>
<td>11.04b</td>
<td>11.64b</td>
<td>11.91b</td>
<td>11.34</td>
<td>3.6</td>
</tr>
<tr>
<td>SK³</td>
<td>18.00c</td>
<td>19.42c</td>
<td>19.44c</td>
<td>19.89c</td>
<td>20.88c</td>
<td>19.53</td>
<td>5.3</td>
</tr>
<tr>
<td>E2⁴</td>
<td>29.30d</td>
<td>27.02d</td>
<td>26.24d</td>
<td>27.22d</td>
<td>29.00d</td>
<td>27.76</td>
<td>4.8</td>
</tr>
</tbody>
</table>

*Each value is a mean of 3 determinations.
**Means followed by same letter within column are not different (p≤0.05).
TS¹ = thin stillage.
E1² = concentrate at end of effect 1, after stage 4 of evaporator.
SK³ = concentrate after oil skimming before enter into stage 8.
E2⁴ = concentrate at end of effect 2, after stage 8 of evaporator.
CV⁵ = coefficient of variation

4.3.1. Effects of evaporator heat treatment on fouling before plant cleaning

For samples collected before cleaning, deposits began to accumulate rapidly indicated by a rapid increase of fouling resistance and with fouling rates (before deposit sloughing) of 0.23 to 0.86 m² K/kW (Table 4.4). Induction periods (IP) were less than 0.083 hr (5 mins) (Fig. 4.3 and
Table 4.4). IP was not a key fouling characteristic for the series of tests because of the rapid increase of fouling resistance (Fig. 4.4). Among the fouling rates (FR1, FR2, FR5 and FRS), FRS would be the best to illustrate fouling behavior prior to deposit sloughing. When sloughing occurred, there was a decrease of fouling resistance (more than 30%), resulting in a decrease in fouling rate (Table 4.4).

Table 4.4. Fouling parameters of thin stillage and concentrates before cleaning

<table>
<thead>
<tr>
<th>Sample</th>
<th>FR1* (m²·K/kW·hr)</th>
<th>FR2* (m²·K/kW·hr)</th>
<th>FR5* (m²·K/kW·hr)</th>
<th>FSP* (hr)</th>
<th>FRS* (m²·K/kW·hr)</th>
<th>R_{max}* (m²·K/kW·hr)</th>
<th>IP* (hr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TS</td>
<td>0.27**</td>
<td>0.17</td>
<td>0.066</td>
<td>2.65</td>
<td>0.23</td>
<td>0.38</td>
<td>0.083</td>
</tr>
<tr>
<td>E1</td>
<td>0.47</td>
<td>0.14</td>
<td>0.067</td>
<td>1.04</td>
<td>0.46</td>
<td>0.43</td>
<td>0.008</td>
</tr>
<tr>
<td>SK</td>
<td>0.55</td>
<td>0.15</td>
<td>0.081</td>
<td>0.87</td>
<td>0.73</td>
<td>0.47</td>
<td>0.008</td>
</tr>
<tr>
<td>E2</td>
<td>0.43</td>
<td>0.14</td>
<td>0.091</td>
<td>0.80</td>
<td>0.86</td>
<td>0.52</td>
<td>0.008</td>
</tr>
</tbody>
</table>

*FR1, FR2, FR5 = fouling rate in 1, 2, 5 hr. FSP = first sloughing point. FRS = fouling rate before first sloughing point. R_{max} = maximum fouling resistance. IP = induction periods

**Mean value of two tests. No differences were detected among all treatment means (P ≤ 0.05)

After reaching maximum fouling resistance, deposits often would fall off the probe surface as was shown by the sudden decrease of fouling resistance in the fouling curve (Fig. 4.3). Deposit sloughing took place about 1 hr after tests started. A higher fouling rate was observed before the sloughing took place for SK and E2 samples compared with TS and E1 samples. Those samples (SK and E2) tended to have more complete sloughing, indicated by fouling resistance values that decreased to less than 0.05 m²·K/kW. TS and E1 samples had lower maximum fouling resistances compared with SK and E2 samples (Table 4.4).
Figure 4.3. Fouling resistance of thin stillage and evaporator concentrates vs time before cleaning. TS-1/2= thin stillage samples collected on the first and second batch, E1-1/2= samples from end of effect 1 (after stage 4), collected on the first and second batches, SK-1/2= samples after skimming (before stage 8), collected on the first and second batches, E2-1/2= samples at end of effect 2 (after stage 8), collected on the first and second batches.

We observed a faster fouling rate and more severe fouling curve with more heat treatment as seen in SK and E2 fouling profiles compared with TS and E1 samples from the mean fouling resistance vs time (Fig. 4.4). However, there were no differences ($P \leq 0.05$) in fouling rates, maximum fouling resistance or induction periods among the treatments (Table 4.4).
Figure 4.4. Mean (means of two observations) fouling resistance vs time of samples before plant shut cleaning. TS = thin stillage samples collected on the first and second batches, E1 = samples from end of effect 1 (after stage 4), SK = samples after skimming (before stage 8), E2 = samples at end of effect 2 (after stage 8).

4.3.2. Effects of evaporator heat treatment of thin stillage on fouling after plant cleaning

$R_f$ increased throughout the test period. Fouling curve of sample tested after cleaning was linear ($R^2 = 0.98 \pm 0.017$) while the fouling curve of sample tested before cleaning was less linear ($R^2 = 0.73 \pm 0.020$). Sloughing rarely was observed in these experiments. Only one sloughing point was seen during all the fouling tests after cleaning (Fig. 4.5).
Figure 4.5. Fouling resistance of thin stillage and concentrates vs time after cleaning. (a) batch 3 sample (a wk after cleaning), (b) batch 4 sample (2 wk after cleaning), (c) batch 5 sample. (3 wk after cleaning)

\( R_f \) increased throughout the test period. Fouling curve of sample tested after cleaning was linear \( (R^2 = 0.98 \pm 0.017) \) while the fouling curve of sample tested before cleaning was less linear \( (R^2 = 0.73 \pm 0.020) \). Sloughing rarely was observed in these experiments. Only one
The sloughing point was seen during all the fouling tests after cleaning (Fig. 4.5).

![Graph showing fouling curve after cleaning](image)

**Figure 4.6.** Example of linear regression fit to fouling curve after cleaning. (a) fouling rate in 5 hr; (b) fouling rate in 2 hr

Fouling rates (FR1, FR2, FR5 and SFR) were similar (Table 4.5) because of the linearity of
the fouling curve and a better fit of the linear regression line (Fig. 4.6). Thus FR5 would be a good parameter to illustrate the rate of fouling for this series of tests. From the mean fouling curve (Fig. 4.7), we observed thin stillage had slower accumulation of fouling deposits in the 5 hr tests compared with concentrates (E1, SK and E2). TS mean fouling rate was 0.018 (m²·K/kW·hr) and E2 had a mean fouling rate of 0.066 m²·K/kW·hr (Table 4.5.). Induction periods of TS were 0.65 hr (40 min), while induction periods of concentrates were 0.10 to 0.31 hr (15 to 17 mins) (Table 4.5). Maximum fouling resistance was 0.19 m²·K/kW (Table 4.5) which was lower than R_max before cleaning (0.52 m²·K/kW) (Table 4.4).

![Figure 4.7. Mean fouling resistance vs time for samples after cleaning](image)

Fouling behavior of the samples collected at same locations varied from batch to batch
(Fig. 4.8). An average of more than 50% of variation was observed among batches. E1 samples had the largest level of variation (more than 90% CV for fouling rate and maximum fouling resistance) (Table 4.6). E2 samples had the smallest level of variation (30%) among three batches. There were no differences of fouling rates, maximum fouling resistances and induction periods among treatments.

**Table 4.5 Fouling parameters of thin stillage and concentrates after cleaning.**

<table>
<thead>
<tr>
<th>Sample</th>
<th>FR1* (m²·K/kW·hr)</th>
<th>FR2* (m²·K/kW·hr)</th>
<th>FR5* (m²·K/kW·hr)</th>
<th>FSP* (hr)</th>
<th>FRS* (m²·K/kW·hr)</th>
<th>Rmax* (m²·K/kW)</th>
<th>IP* (hr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TS</td>
<td>0.019</td>
<td>0.020</td>
<td>0.018</td>
<td>&gt;5</td>
<td>0.018</td>
<td>0.089</td>
<td>0.65</td>
</tr>
<tr>
<td>E1</td>
<td>0.06</td>
<td>0.051</td>
<td>0.042</td>
<td>&gt;5</td>
<td>0.042</td>
<td>0.19</td>
<td>0.31</td>
</tr>
<tr>
<td>SK</td>
<td>0.042</td>
<td>0.039</td>
<td>0.030</td>
<td>&gt;5</td>
<td>0.033</td>
<td>0.14</td>
<td>0.27</td>
</tr>
<tr>
<td>E2</td>
<td>0.073</td>
<td>0.064</td>
<td>0.045</td>
<td>&gt;5</td>
<td>0.045</td>
<td>0.19</td>
<td>0.096</td>
</tr>
</tbody>
</table>

*FR1, FR2, FR5 = fouling rates in 1, 2 and 5 hr. FSP = first sloughing point. FRS = fouling rate before first sloughing point. Rmax = maximum fouling resistance. IP = induction period.

**Mean value of three tests. No differences were detected among treatment means (P<0.05).**

**Table 4.6. Mean fouling rate and maximum fouling resistance after cleaning.**

<table>
<thead>
<tr>
<th>Sample</th>
<th>FR5¹ (m²·K/kW·hr)</th>
<th>CV² (%)</th>
<th>Rmax³ (m²·K/kW)</th>
<th>CV (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TS</td>
<td>0.018*</td>
<td>0.78</td>
<td>0.089</td>
<td>77</td>
</tr>
<tr>
<td>E1</td>
<td>0.042</td>
<td>0.93</td>
<td>0.19</td>
<td>91</td>
</tr>
<tr>
<td>Sk</td>
<td>0.030</td>
<td>0.54</td>
<td>0.14</td>
<td>57</td>
</tr>
<tr>
<td>E2</td>
<td>0.045</td>
<td>0.30</td>
<td>0.19</td>
<td>32</td>
</tr>
</tbody>
</table>

¹FR5 = mean fouling rate in 5 hr of 3 batches after cleaning.
²CV = coefficient of variation of three replicates.
³Rmax = maximum fouling resistance.

*No differences were detected among all treatment means (P<0.05). Batch 3 was collected a wk after plant shut down and cleaning. Batch 4 was collected two wk after plant shut down and batch 5 was 4 wk. No correlations among samples collection time.
and fouling behavior were observed.

Figure 4.8. Fouling behavior among batches after cleaning. (a), (b), (c), (d), fouling curves of TS, E1, SK and E2 collected from different batches, respectively
4.3.3. Comparisons of fouling characteristics before and after plant cleaning

Fouling rates and maximum fouling resistances of all four samples decreased after the plant cleaning (Table 4.7). Fouling curves of samples were more linear \((0.96 \leq R^2 \leq 0.99)\) after cleaning than before cleaning \((0.71 \leq R^2 \leq 0.76)\). There were less sloughing points after plant cleaning \((SP=1)\) than before plant cleaning \((SP=10)\).

Fouling rates before deposit sloughing for each sample at the same location were different before and after plant shut down and cleaning (Table 4.7). Maximum fouling resistances of TS, SK and E2 samples were different before and after plant shut down and cleaning (Table 4.7). Both fouling rates and maximum fouling resistances decreased after plant cleaning (Fig.4.9). SFR decreased by more than 90% after plant shut down and cleaning. \(R_{\text{max}}\) decreased by more than 50% after plant shut down and cleaning (Table 4.7). FR1, FR2 and FR5 decreased after plant cleaning at each sample location, while IP increased (A.3.2.2). Cleaning is a factor influencing fouling characteristics.

Table 4.7. Comparison of fouling rate and maximum fouling resistance before and after cleaning.

<table>
<thead>
<tr>
<th></th>
<th>TS</th>
<th>EI</th>
<th>SK</th>
<th>E2</th>
</tr>
</thead>
<tbody>
<tr>
<td>FRS before(^1) ((\text{m}^2 \cdot \text{K}/\text{kW} \cdot \text{hr}))</td>
<td>0.23 ± 0.19b</td>
<td>0.46 ± 0.041b</td>
<td>0.73 ± 0.28b</td>
<td>0.86 ± 0.047b</td>
</tr>
<tr>
<td>FRS after(^1) ((\text{m}^2 \cdot \text{K}/\text{kW} \cdot \text{hr}))</td>
<td>0.018 ± 0.014a</td>
<td>0.042 ± 0.039a</td>
<td>0.033 ± 0.020a</td>
<td>0.045 ± 0.013a</td>
</tr>
<tr>
<td>(R_{\text{max}}) before(^2) ((\text{m}^2 \cdot \text{K}/\text{kW}))</td>
<td>0.38 ± 0.058b</td>
<td>0.43 ± 0.012ab</td>
<td>0.47 ± 0.026b</td>
<td>0.52 ± 0.019b</td>
</tr>
<tr>
<td>(R_{\text{max}}) after(^2) ((\text{m}^2 \cdot \text{K}/\text{kW}))</td>
<td>0.088 ± 0.068a</td>
<td>0.20 ± 0.17a</td>
<td>0.11 ± 0.08a</td>
<td>0.17 ± 0.06a</td>
</tr>
</tbody>
</table>

\(^1\)FRS before / after = fouling rate after deposit sloughing  
\(^2\)\(R_{\text{max}}\) = maximum fouling resistance  
Value with the same letter in the same column were not different \((P<0.05)\)
Figure 4.9. Maximum fouling resistance ($R_{\text{max}}$) and fouling rate before sloughing (FRS) of samples from different locations before and after cleaning. Bar in the same letter in the same column were not different (P<0.05)

Fouling rates before deposit sloughing, maximum fouling resistance and induction periods before and after plant cleaning in this study were compared with previous studies (Table 4.8).
Fouling rates were calculated differently. Challa et al (2017) and Wilkins et al (2006a) defined fouling rate as the slope of the regression line ($R_t$ vs. $t$) from $t = 0$ to $t = 5$ hr. They were not setting intercept as 0. Mean fouling rate of samples before cleaning ($0.23 \text{ m}^2\cdot\text{K/kW}\cdot\text{hr}$) was higher than previous studies. Mean fouling rate of samples after cleaning was lower than previous studies. Challa et al (2017) also observed changes of fouling behavior of thin stillage and concentrates after plant cleaning. They reported that induction periods were longer than 5 hr for thin stillage and concentrates were collected after cleaning.

Table 4.8. Comparison of thin stillage fouling rate and maximum fouling resistance with previous research.

<table>
<thead>
<tr>
<th></th>
<th>Present study before$^1$</th>
<th>Present study after$^1$</th>
<th>Challa (2014) before$^1$</th>
<th>Challa (2014) after$^1$</th>
<th>Wilkins et al (2006a)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean fouling rate</td>
<td>0.23</td>
<td>0.018</td>
<td>0.027</td>
<td>NA</td>
<td>0.046 to 0.062</td>
</tr>
<tr>
<td>(m$^2\cdot$K/kW/hr)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Maximum fouling</td>
<td>0.38</td>
<td>0.089</td>
<td>0.15</td>
<td>NA</td>
<td>0.4 ± 0.05</td>
</tr>
<tr>
<td>resistance (m$^2\cdot$K/kW)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Induction period (hr)</td>
<td>0.083</td>
<td>0.65</td>
<td>NA</td>
<td>&gt; 5</td>
<td>0.42 to 2.4</td>
</tr>
</tbody>
</table>

$^1$ before and after plant shut down and cleaning.

4.4. Conclusions

Commercial thin stillage and evaporator concentrates from a multiple effect evaporator were used to study effects of evaporator heat treatment and plant cleaning on thin stillage fouling characteristics. Large variations of fouling parameters among batches were observed for samples collected both before and after plant cleaning. No differences among the different heat treatment samples were detected.

Plant cleaning had effects on fouling characteristics of thin stillage and concentrates.
Fouling rate and maximum fouling resistance decreased and induction period increased for samples collected after plant cleaning. We concluded that plant shut down and cleaning reduce fouling in general. Future work could study changes of thin stillage composition after plant cleaning that lead to fouling.
CHAPTER 5. RECOMMENDATIONS

1. Present fouling experiments did not include analysis on fouling deposits due to the difficulty of removing deposits without damaging the probe. Methods could be developed to safely remove and collect fouling deposits after each test. Analysis of deposit thickness, compositions and distribution will help us understand the process of deposits formation and study fouling mechanisms.

2. In chapters 4 and 5, variations of fouling behaviors among batches were observed. A further analysis of thin stillage composition of each batch and compare them with fouling parameters in the tests could help us study effects of composition on thin stillage fouling.

3. Model thin stillage of 1% starch was used in this study as a first step model fluid similar to thin stillage that offered repetitive fouling results and a benchmark of analyzing fouling characteristics. Model fluid that has properties and compositions closer to thin stillage could be developed to study thin stillage fouling. For example, protein, phosphate, glycerol and minerals can be added to the model thin stillage.

4. Simulation modeling could be performed to back up experiment results and explain the reason of certain fouling behavior.

5. Cleaning of thin stillage fouling deposits could be examined by doing tests during the cleaning process after each fouling test to monitor the mitigation of fouling deposits.

6. A portable fouling apparatus could be developed to monitor fouling process in the evaporators of commercial plants.

7. Pilot scale evaporators and fouling apparatus could be developed.
LITERATURE CITED:


APPENDIX A. FOULING TEST PROCEDURE

1. Check the annular fouling probe for leaks by circulating hot tap water through the probe into the tank.

2. Clean the fouling system by recirculating 10 L 1% (w/v) Alconox solution for 20 min followed by a 150 L hot water rinse.

3. Connect the thermocouple extensions (male) from the data logger to the female ends of the fouling probe.

4. Charge the tank with 7 L of testing fluids.

5. Start the heat exchanger and adjust the temperature to desired bulk temperature.

6. Turn on the pump connected to the heating fluid tank to recirculate through the heat exchanger.

7. Recirculate tap water from the tank, through the heat exchanger, fouling probe and back into the tank.

8. Wait until the heat exchanger fluid and the test fluid reach equilibrium.

9. Slowly add 70 g of starch into the tank (if the test fluid is 1% starch). Recirculate the 1% (w/v) starch slurry from the tank through the heat exchanger, fouling probe and back into the tank.

10. Collect a 500 ml sample for viscosity, density and pH measurement.

11. Turn on the data logger, set the parameters.

12. Turn on the power to the 220 V power supply to the fouling apparatus and adjust the average surface temperature on probe to the desired initial probe temperature (80, 100 and 120°C) by turning the knob connected to limit controller.

13. Start the data logger and collect data for the test fluid.

14. After 5 h end the experiment by disconnecting the 220 V power to the probe, turn off the heat exchanger.
15. Collect a 500 mL sample from the tank for analysis.

16. Discard the test fluid into drain and empty the tank.

17. Scrub the tank walls with brush and rinse the tank with hot tap water.

18. Remove the thermocouple connections and dismantle the annular probe. Scrape the fouling deposits with a plastic spatula into a sampling vial and store at 4°C.

19. Soak the probe overnight in 5% (w/v) NaOH, use 1000 mL tap water to rinse the probe to clean remove all NaOH before use.
# APPENDIX B. FOULING APPARATUS

Table B.1. Fouling equipment specifications

<table>
<thead>
<tr>
<th>Equipment</th>
<th>Specifications</th>
</tr>
</thead>
<tbody>
<tr>
<td>Centrifugal Pump</td>
<td>Model: S-115 RZ, Company: Iwaki Walchem, Iwaki, Japan</td>
</tr>
<tr>
<td></td>
<td>115V 60Hz 3.0A</td>
</tr>
<tr>
<td>Data logger</td>
<td>Model: RDXL 120, Company: Omega, One Omega Drive,</td>
</tr>
<tr>
<td></td>
<td>Stanford, CT.</td>
</tr>
<tr>
<td>Annular probe</td>
<td>Model: FIREROD 1025</td>
</tr>
<tr>
<td></td>
<td>Product No. SJ24AX-2835</td>
</tr>
</tbody>
</table>
APPENDIX C. THIN STILLAGE SAMPLE INFORMATION

Table C.1. Sample collection dates of commercial thin stillage for temperature effects study

<table>
<thead>
<tr>
<th>Batch number</th>
<th>Date</th>
<th>Note</th>
</tr>
</thead>
<tbody>
<tr>
<td>Batch 1</td>
<td>01.15.2015</td>
<td>A day after plant cleaning</td>
</tr>
<tr>
<td>Batch 2</td>
<td>01.29.2015</td>
<td>A day after plant cleaning</td>
</tr>
<tr>
<td>Batch 3</td>
<td>02.19.2015</td>
<td>On the day of plant cleaning</td>
</tr>
</tbody>
</table>

Table C.2. Treatments conditions in commercial thin stillage fouling tests.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Total solids (% db)</th>
<th>Viscosity (cP)</th>
<th>Reynold number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Batch 1</td>
<td>7.23 ± 0.33</td>
<td>19.00 ± 0.25</td>
<td>460 ± 20</td>
</tr>
<tr>
<td>Batch 2</td>
<td>7.02 ± 0.26</td>
<td>18.00 ± 0.50</td>
<td>480 ± 20</td>
</tr>
<tr>
<td>Batch 3</td>
<td>6.97 ± 0.23</td>
<td>16.23 ± 0.46</td>
<td>520 ± 20</td>
</tr>
</tbody>
</table>
APPENDIX D. DATA ANALYSIS

D.1. Regression analysis

Regression analysis by directly fitting a linear regression line and displaying $R^2$ using Microsoft Excel 2013 resulted in negative $R^2$ value (Fig. A.1). This problem could be solved by using regression analysis on the $R_f$ vs. time raw data (Table A.3).

Figure D.1. Example of regression analyses by Microsoft Excel 2013.
Table D.1. Example of regression analyses for $R^2$ calculation by Microsoft Excel 2013.

<table>
<thead>
<tr>
<th>Regression Statistics</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Multiple R</td>
<td>0.8949217</td>
</tr>
<tr>
<td>R Square</td>
<td>0.8008848</td>
</tr>
<tr>
<td>Adjusted R Square</td>
<td>0.7975515</td>
</tr>
<tr>
<td>Standard Error</td>
<td>0.0884798</td>
</tr>
<tr>
<td>Observations</td>
<td>301</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>ANOVA</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>df</td>
<td>SS</td>
</tr>
<tr>
<td>Regression</td>
<td>1</td>
</tr>
<tr>
<td>Residual</td>
<td>300</td>
</tr>
<tr>
<td>Total</td>
<td>301</td>
</tr>
</tbody>
</table>

D.2. ANOVA analyses

1. Temperature effects on commercial thin stillage
```r
> datal=read.csv(file.choose(),header=T)
> show(datal)

<table>
<thead>
<tr>
<th>Group</th>
<th>FR1</th>
<th>FR2</th>
<th>FR5</th>
<th>Rmax</th>
<th>IP</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>a</td>
<td>0.4306</td>
<td>0.2183</td>
<td>0.0477</td>
<td>0.50746894</td>
</tr>
<tr>
<td>2</td>
<td>a</td>
<td>0.3976</td>
<td>0.2958</td>
<td>0.0691</td>
<td>0.47152618</td>
</tr>
<tr>
<td>3</td>
<td>a</td>
<td>0.3210</td>
<td>0.2450</td>
<td>0.0980</td>
<td>0.43118315</td>
</tr>
<tr>
<td>4</td>
<td>b</td>
<td>0.0583</td>
<td>0.0374</td>
<td>0.0234</td>
<td>0.10226036</td>
</tr>
<tr>
<td>5</td>
<td>b</td>
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<td>-0.0003</td>
<td>0.0009</td>
<td>0.01217323</td>
</tr>
<tr>
<td>6</td>
<td>b</td>
<td>0.0100</td>
<td>0.0073</td>
<td>0.0041</td>
<td>0.02017166</td>
</tr>
<tr>
<td>7</td>
<td>c</td>
<td>0.0332</td>
<td>0.0327</td>
<td>0.0476</td>
<td>0.37801362</td>
</tr>
<tr>
<td>8</td>
<td>c</td>
<td>0.0240</td>
<td>0.0223</td>
<td>0.0296</td>
<td>0.20649796</td>
</tr>
<tr>
<td>9</td>
<td>c</td>
<td>0.0103</td>
<td>0.0124</td>
<td>0.0641</td>
<td>0.49541030</td>
</tr>
<tr>
<td>10</td>
<td>d</td>
<td>0.0090</td>
<td>0.0045</td>
<td>0.0025</td>
<td>0.01495037</td>
</tr>
<tr>
<td>11</td>
<td>d</td>
<td>-0.0079</td>
<td>-0.0033</td>
<td>0.0022</td>
<td>0.02010393</td>
</tr>
<tr>
<td>12</td>
<td>d</td>
<td>0.0038</td>
<td>0.0043</td>
<td>0.0075</td>
<td>0.04997015</td>
</tr>
</tbody>
</table>

> FRI=aim(FRI~Group,data=datal)
> summary(FRI)

```

```

```

---
Signif. codes: 0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1

> TukeyHSD(FRI)

Tukey multiple comparisons of means
95% family-wise confidence level

Fit: aov(formula = FRI ~ Group, data = datal)

```r
> Group

diff  lwr   upr  p adj
b-a -0.361600000 -0.4486642 -0.27453577 0.0000046
c-a -0.3605666667 -0.4476309 -0.27350244 0.0000047
d-a -0.381433333 -0.4684976 -0.29436910 0.0000031
c-b 0.010333333 -0.0860309 0.08809756 0.9999785
d-b -0.019833333 -0.1068976 0.06723090 0.8826511
d-c -0.0208666667 -0.1079309 0.06619756 0.8668466
```
> plot(TukeyHSD(FR1))
> data1=read.csv(file.choose(),header=T)
> show(data1)

<table>
<thead>
<tr>
<th>Group</th>
<th>FR1</th>
<th>FR2</th>
<th>FR5</th>
<th>Rmax</th>
<th>IP</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>a</td>
<td>0.4306</td>
<td>0.2183</td>
<td>0.0477</td>
<td>0.50746894</td>
</tr>
<tr>
<td>2</td>
<td>a</td>
<td>0.3976</td>
<td>0.2958</td>
<td>0.0691</td>
<td>0.47152618</td>
</tr>
<tr>
<td>3</td>
<td>a</td>
<td>0.3210</td>
<td>0.2450</td>
<td>0.0980</td>
<td>0.43118315</td>
</tr>
<tr>
<td>4</td>
<td>b</td>
<td>0.0563</td>
<td>0.0374</td>
<td>0.0234</td>
<td>0.10226036</td>
</tr>
<tr>
<td>5</td>
<td>b</td>
<td>-0.0039</td>
<td>-0.0003</td>
<td>0.0009</td>
<td>0.01217323</td>
</tr>
<tr>
<td>6</td>
<td>b</td>
<td>0.0100</td>
<td>0.0073</td>
<td>0.0041</td>
<td>0.02017166</td>
</tr>
<tr>
<td>7</td>
<td>c</td>
<td>0.0332</td>
<td>0.0327</td>
<td>0.0476</td>
<td>0.37801362</td>
</tr>
<tr>
<td>8</td>
<td>c</td>
<td>0.0240</td>
<td>0.0223</td>
<td>0.0296</td>
<td>0.20649796</td>
</tr>
<tr>
<td>9</td>
<td>c</td>
<td>0.0103</td>
<td>0.0124</td>
<td>0.0641</td>
<td>0.49541030</td>
</tr>
<tr>
<td>10</td>
<td>d</td>
<td>0.0090</td>
<td>0.0045</td>
<td>0.0025</td>
<td>0.01495037</td>
</tr>
<tr>
<td>11</td>
<td>d</td>
<td>-0.0079</td>
<td>-0.0033</td>
<td>0.0022</td>
<td>0.02010393</td>
</tr>
<tr>
<td>12</td>
<td>d</td>
<td>0.0038</td>
<td>0.0043</td>
<td>0.0075</td>
<td>0.04997015</td>
</tr>
</tbody>
</table>

> FR1=aov(FR1~Group,data=data1)
> summary(FR1)

<table>
<thead>
<tr>
<th>Df</th>
<th>Sum Sq</th>
<th>Mean Sq</th>
<th>F value</th>
<th>Pr(&gt;F)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group</td>
<td>3</td>
<td>0.30531</td>
<td>0.10177</td>
<td>91.79</td>
</tr>
<tr>
<td>Residuals</td>
<td>8</td>
<td>0.00887</td>
<td>0.00111</td>
<td>-</td>
</tr>
</tbody>
</table>

---
Signif. codes: 0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1

> TukeyHSD(FR1)

Tukey multiple comparisons of means
95% family-wise confidence level

Fit: aov(formula = FR1 ~ Group, data = data1)

$Group

<table>
<thead>
<tr>
<th>diff</th>
<th>lwr</th>
<th>upr</th>
<th>p adj</th>
</tr>
</thead>
<tbody>
<tr>
<td>b-a</td>
<td>-0.361600000</td>
<td>-0.4486642</td>
<td>-0.27453577</td>
</tr>
<tr>
<td>c-a</td>
<td>-0.360566667</td>
<td>-0.4476309</td>
<td>-0.27350244</td>
</tr>
<tr>
<td>d-a</td>
<td>-0.381433333</td>
<td>-0.4684976</td>
<td>-0.29436910</td>
</tr>
<tr>
<td>c-b</td>
<td>0.001033333</td>
<td>-0.0860309</td>
<td>0.08509756</td>
</tr>
<tr>
<td>d-b</td>
<td>0.019033333</td>
<td>-0.1068976</td>
<td>0.06723090</td>
</tr>
<tr>
<td>d-c</td>
<td>0.020866667</td>
<td>-0.1079309</td>
<td>0.06619756</td>
</tr>
</tbody>
</table>

> FR2=aov(FR2~Group,data=data1)
> summary(FR2)

<table>
<thead>
<tr>
<th>Df</th>
<th>Sum Sq</th>
<th>Mean Sq</th>
<th>F value</th>
<th>Pr(&gt;F)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group</td>
<td>3</td>
<td>0.13025</td>
<td>0.04342</td>
<td>83.89</td>
</tr>
<tr>
<td>Residuals</td>
<td>8</td>
<td>0.00414</td>
<td>0.00052</td>
<td>-</td>
</tr>
</tbody>
</table>

---
Signif. codes: 0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1
> TukeyHSD(FR2)
  Tukey multiple comparisons of means
  95% family-wise confidence level

Fit: aov(formula = FR2 ~ Group, data = data1)

$Group
  diff  lwr  upr  p adj
b-a  -0.23823333 -0.29771855 -0.17874812  0.0000061
c-a  -0.23056667 -0.29005188 -0.17108145  0.0000078
d-a  -0.25120000 -0.31068521 -0.19171479  0.0000041
c-b   0.00766667  0.05181855  0.06715188  0.9747368
d-b  -0.01296667 -0.07245188  0.04651855  0.8950270
d-c  -0.02063333 -0.08011855  0.03885188  0.6934716

> FR5=aov(FR5~Group,data=data1)
> summary(FR5)

    Df Sum Sq Mean Sq F value Pr(>F)
Group    3  0.009239  0.003080  11.28   0.00303 **
Residuals 8  0.002184  0.000273

---
Signif. codes:  0 '***' 0.001 '**' 0.01 '*' 0.05 '.' 0.1 ' ' 1

> TukeyHSD(FR5)
  Tukey multiple comparisons of means
  95% family-wise confidence level

Fit: aov(formula = FR5 ~ Group, data = data1)

$Group
  diff  lwr  upr  p adj
b-a   -0.06213333 -0.105335160 -0.0189315066  0.0075527
c-a   -0.02450000 -0.067701827  0.0187018267  0.3330616
d-a   -0.06753333 -0.110735160 -0.0243315066  0.0045912
c-b   0.03763333  0.00556493  0.0698351601  0.0895350
d-b  -0.00540000 -0.048601827  0.0378018267  0.9768391
d-c  -0.04303333 -0.086235160  0.0001684934  0.0508866

> Rmax=aov(Rmax~Group,data=data1)
> summary(Rmax)

    Df Sum Sq Mean Sq F value Pr(>F)
Group    3  0.4482  0.14939  23.52   0.000254 ***
Residuals 8  0.0508  0.00635

---
Signif. codes:  0 '***' 0.001 '**' 0.01 '*' 0.05 '.' 0.1 ' ' 1
```r
> Rmax=aoe(Rmax~Group, data=da1)
> summary(Rmax)

    Df Sum Sq Mean Sq F value Pr(>F)
Group     3 0.4482  0.14939  23.52 0.000254 ***
Residuals 8 0.0508  0.00635
---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1

> TukeyHSD(Rmax)

Tukey multiple comparisons of means
  95% family-wise confidence level

Fit: aov(formula = Rmax ~ Group, data = da1)

$Group
diff  lwr  upr    p adj
b-a  -0.42519101 -0.6335983 -0.21678369 0.0009237
 c-a  -0.11008546 -0.3184928  0.09832185 0.3868672
 d-a  -0.44171794 -0.6501253 -0.23331063 0.0006354
 c-b   0.31510554  0.1066982  0.52351286 0.0056166
 d-b  -0.01652693 -0.2249342  0.19188038 0.9937938
 d-c  -0.33163248 -0.5400398 -0.12322516 0.0041171

> IP=aoe(IP~Group, data=da1)
> summary(IP)

    Df Sum Sq Mean Sq F value Pr(>F)
Group     3 41.45 13.818 12.19  0.00236 **
Residuals 8  9.07  1.134
---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1

> TukeyHSD(IP)

Tukey multiple comparisons of means
  95% family-wise confidence level

Fit: aov(formula = IP ~ Group, data = da1)

$Group
diff  lwr  upr    p adj
b-a   3.674333  0.8902848 6.4583819 0.0123288
 c-a  1.697667 -1.0863819 4.4817152 0.2805929
 d-a  4.857667  2.0736181 7.6417152 0.0023090
 c-b -1.976667 -4.7607152  0.8073819 0.1835617
 d-b  1.183333 -1.6007152 3.9673819 0.5540187
 d-c  3.160000  0.3759514 5.9440486 0.0273647
```
> dataTS=read.csv(file.choose(),header=T)
> show(dataTS)

<table>
<thead>
<tr>
<th></th>
<th>Tb</th>
<th>Ti</th>
<th>Dt</th>
<th>FR1</th>
<th>FR2</th>
<th>FR5</th>
<th>Rmax</th>
<th>IP</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>80</td>
<td>120</td>
<td>40</td>
<td>0.4306</td>
<td>0.2183</td>
<td>0.0477</td>
<td>0.50746894</td>
<td>0.330</td>
</tr>
<tr>
<td>2</td>
<td>80</td>
<td>120</td>
<td>40</td>
<td>0.3976</td>
<td>0.2958</td>
<td>0.0691</td>
<td>0.47152618</td>
<td>0.067</td>
</tr>
<tr>
<td>3</td>
<td>80</td>
<td>120</td>
<td>40</td>
<td>0.3210</td>
<td>0.2450</td>
<td>0.0980</td>
<td>0.43118315</td>
<td>0.030</td>
</tr>
<tr>
<td>4</td>
<td>80</td>
<td>100</td>
<td>20</td>
<td>0.0583</td>
<td>0.0374</td>
<td>0.0234</td>
<td>0.10226036</td>
<td>1.450</td>
</tr>
<tr>
<td>5</td>
<td>80</td>
<td>100</td>
<td>20</td>
<td>-0.0039</td>
<td>-0.0003</td>
<td>0.0009</td>
<td>0.01217323</td>
<td>5.000</td>
</tr>
<tr>
<td>6</td>
<td>80</td>
<td>100</td>
<td>20</td>
<td>0.0100</td>
<td>0.0073</td>
<td>0.0041</td>
<td>0.02017166</td>
<td>5.000</td>
</tr>
<tr>
<td>7</td>
<td>60</td>
<td>120</td>
<td>60</td>
<td>0.0332</td>
<td>0.0327</td>
<td>0.0476</td>
<td>0.37801362</td>
<td>1.200</td>
</tr>
<tr>
<td>8</td>
<td>60</td>
<td>120</td>
<td>60</td>
<td>0.0240</td>
<td>0.0223</td>
<td>0.0296</td>
<td>0.20649796</td>
<td>2.150</td>
</tr>
<tr>
<td>9</td>
<td>60</td>
<td>120</td>
<td>60</td>
<td>0.0103</td>
<td>0.0124</td>
<td>0.0641</td>
<td>0.49541030</td>
<td>2.170</td>
</tr>
<tr>
<td>10</td>
<td>60</td>
<td>100</td>
<td>40</td>
<td>0.0090</td>
<td>0.0045</td>
<td>0.0025</td>
<td>0.01495037</td>
<td>5.000</td>
</tr>
<tr>
<td>11</td>
<td>60</td>
<td>100</td>
<td>40</td>
<td>-0.0079</td>
<td>-0.0033</td>
<td>0.0022</td>
<td>0.02010393</td>
<td>5.000</td>
</tr>
<tr>
<td>12</td>
<td>60</td>
<td>100</td>
<td>40</td>
<td>0.0038</td>
<td>0.0043</td>
<td>0.0075</td>
<td>0.04997015</td>
<td>5.000</td>
</tr>
</tbody>
</table>

> FR1TWO=lm(FR1~Tb+Ti,data=dataTS)
> summary(FR1TWO)

    Df Sum Sq Mean Sq   F value Pr(>F)    
Tb     1 0.10853 0.10853 10.18    0.0110  * 
Ti     1 0.10971 0.10971 10.29    0.0107  * 
Residuals   9 0.09594 0.01066

---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1

> FR1TWO=lm(FR1~Tb+Ti,data=dataTS)
> summary(FR1TWO)

    Df Sum Sq Mean Sq   F value Pr(>F)    
Tb     1 0.10853 0.10853 10.18    0.0110  * 
Ti     1 0.10971 0.10971 10.29    0.0107  * 
Residuals   9 0.09594 0.01066

---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1

> FR2TWO=lm(FR2~Tb+Ti,data=dataTS)
> summary(FR2TWO)

    Df Sum Sq Mean Sq   F value Pr(>F)    
Tb     1 0.04448 0.04448 10.10    0.01123 * 
Ti     1 0.05026 0.05026 11.41    0.00816 ** 
Residuals   9 0.03965 0.00441

---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1

> FR2TWO=lm(FR2~Tb+Ti+Tb:Ti,data=dataTS)
> summary(FR2TWO)

    Df Sum Sq Mean Sq   F value Pr(>F)    
Tb     1 0.04448 0.04448 85.94    1.49e-05 *** 
Ti     1 0.05026 0.05026 97.11    9.47e-06 *** 
Tb:Ti  1 0.03551 0.03551 68.61    3.40e-05 *** 
Residuals   8 0.00414 0.00052

---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1

83
```
> FR5Two=aoV(FR5~Tb+Ti,data=dataTS)
> summary(FR5Two)

    Df Sum Sq Mean Sq  F value   Pr(>F)
  Tb     1 0.000671 0.000671 2.4550 0.151557
  Ti     1 0.008295 0.008295 30.3780 0.000375 ***
Residuals 9 0.002458 0.000273
---
Signif. codes: 0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ’.’ 0.1 ‘ ’ 1

> FR5iTwo=aoV(FR5~Tb+Ti,Tb:Ti,data=dataTS)
> summary(FR5iTwo)

    Df Sum Sq Mean Sq  F value   Pr(>F)
  Tb     1 0.000671 0.000671 2.4560 0.155704
  Ti     1 0.008295 0.008295 30.3850 0.000566 ***
  Tb:Ti  1 0.000274 0.000274 1.0020 0.346085
Residuals 8 0.002184 0.000273
---
Signif. codes: 0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ’.’ 0.1 ‘ ’ 1

> RmaxTwo=aoV(Rmax~Tb+Ti,data=dataTS)
> summary(RmaxTwo)

    Df Sum Sq Mean Sq  F value   Pr(>F)
  Tb     1 0.01200 0.01200 1.8860 0.2030
  Ti     1 0.42960 0.42960 67.3700 1.8e-05 ***
Residuals 9 0.05740 0.0064
---
Signif. codes: 0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ’.’ 0.1 ‘ ’ 1

> RmaxiTwo=aoV(Rmax~Tb+Ti,Tb:Ti,data=dataTS)
> summary(RmaxiTwo)

    Df Sum Sq Mean Sq  F value   Pr(>F)
  Tb     1 0.01200 0.01200 1.8920 0.2060
  Ti     1 0.42960 0.42960 67.6190 3.5e-05 ***
  Tb:Ti  1 0.00660 0.00660 1.0330 0.3390
Residuals 8 0.05080 0.0064
---
Signif. codes: 0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ’.’ 0.1 ‘ ’ 1

> IPTwo=aoV(IP~Tb+Ti,data=dataTS)
> summary(IPTwo)

    Df Sum Sq Mean Sq  F value   Pr(>F)
  Tb     1 6.2300 6.2300 6.0450 0.036238 *
  Ti     1 35.0300 35.0300 34.0170 0.000249 ***
Residuals 9 9.2700 1.030
---
Signif. codes: 0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ’.’ 0.1 ‘ ’ 1

> IPTwo=aoV(IP~Tb+Ti,Tb:Ti,data=dataTS)
> summary(IPTwo)

    Df Sum Sq Mean Sq  F value   Pr(>F)
  Tb     1 6.2300 6.2300 5.4910 0.047175 *
  Ti     1 35.0300 35.0300 30.8990 0.000535 ***
  Tb:Ti  1 0.2000 0.2000 0.1750 0.686708
Residuals 8 9.0700 1.130
---
Signif. codes: 0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ’.’ 0.1 ‘ ’ 1
```
2. Temperature effects on model thin stillage

```r
> data1=read.csv(file.choose(),header=T)
> str(data1)
'data.frame': 12 obs. of 7 variables:
  $ Group : Factor w/ 4 levels "a","b","c","d": 1 2 3 4 5 6 7 8
  $ FR1   : num  1.0000 0.8100 0.9300 0.0450 -0.0047 0.0061 0.0220 0.0067 0.0160 0.0051
  $ FR2   : num  0.4830 0.4419 0.3442 0.1819 0.1984 0.0120 0.0110 0.0002 0.0059 0.0035
  $ FR5   : num  0.1416 0.1213 0.1131 0.0860 0.1014 0.0027 0.0023 -0.0037 0.0034 0.0038
  $ Rmax  : num  0.7746 0.6346 0.7217 0.3277 0.3837 0.0309 0.0201 0.0085 0.0190 0.0186
  $ IP    : num  0.0500 0.1167 0.0830 0.8500 0.9500 5.0000 5.0000 5.0000 5.0000 5.0000

> FL1=asov(FR1~Group,data=data1)
> summary(FL1)
Df Sum Sq Mean Sq F value    Pr(>F)
Group     3 1.8371  0.6124  236.5 3.79e-08 ***
Residuals 8  0.0207  0.0026
---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1
> TukeyHSD(FL1)
Tukey multiple comparisons of means
95% family-wise confidence level

Fit: aov(formula = FR1 ~ Group, data = data1)

$Group
  diff     lwr     upr p adj
b-a 0.90466667 -1.0376282  0.7715051 0.0000001
b-c 0.901733333  0.0347949 -0.8686718 0.0000001
d-a 0.904466667 -1.0375282  0.7714051 0.0000001
b-d 0.002833333 0.1302282  0.1358949 0.9998761
d-d 0.000100000 0.1329615  0.1331615 1.0000000
d-c 0.002733333 0.1357949  0.1303282 0.9998888

> FR2=asov(FR2~Group,data=data1)
> summary(FR2)
Df Sum Sq Mean Sq  F value    Pr(>F)
Group     3 0.3504 0.11679  84.12 2.17e-06 ***
Residuals 8 0.0111 0.00139
---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1
```
> TukeyHSD(FR2)
Tukey multiple comparisons of means
95% family-wise confidence level

Fit: aov(formula = FR2 ~ Group, data = data1)

<table>
<thead>
<tr>
<th>Group</th>
<th>diff</th>
<th>lwr</th>
<th>upr</th>
<th>p adj</th>
</tr>
</thead>
<tbody>
<tr>
<td>b-a</td>
<td>-0.243800000</td>
<td>-0.3412258</td>
<td>-0.14637417</td>
<td>0.0001989</td>
</tr>
<tr>
<td>c-a</td>
<td>-0.415300000</td>
<td>-0.5127258</td>
<td>-0.31787417</td>
<td>0.0000038</td>
</tr>
<tr>
<td>d-a</td>
<td>-0.418633333</td>
<td>-0.5160592</td>
<td>-0.32120751</td>
<td>0.0000036</td>
</tr>
<tr>
<td>c-b</td>
<td>-0.171500000</td>
<td>-0.2689258</td>
<td>-0.07407417</td>
<td>0.0021817</td>
</tr>
<tr>
<td>d-b</td>
<td>-0.174833333</td>
<td>-0.2722592</td>
<td>-0.07740751</td>
<td>0.0019270</td>
</tr>
<tr>
<td>d-c</td>
<td>-0.003333333</td>
<td>-0.1007592</td>
<td>0.09409249</td>
<td>0.9994880</td>
</tr>
</tbody>
</table>

> FR5=aov(FR5~Group,data=data1)
> summary(FR5)

<table>
<thead>
<tr>
<th>Df</th>
<th>Sum Sq</th>
<th>Mean Sq</th>
<th>F value</th>
<th>Pr(&gt;F)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group</td>
<td>3</td>
<td>0.031608</td>
<td>0.010536</td>
<td>18.31 0.000609 ***</td>
</tr>
<tr>
<td>Residuals</td>
<td>8</td>
<td>0.004603</td>
<td>0.000575</td>
<td></td>
</tr>
</tbody>
</table>

---
Signif. codes: 0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1

> TukeyHSD(FR5)
Tukey multiple comparisons of means
95% family-wise confidence level

Fit: aov(formula = FR5 ~ Group, data = data1)

<table>
<thead>
<tr>
<th>Group</th>
<th>diff</th>
<th>lwr</th>
<th>upr</th>
<th>p adj</th>
</tr>
</thead>
<tbody>
<tr>
<td>b-a</td>
<td>-0.057533333</td>
<td>-0.12025022</td>
<td>0.0051835495</td>
<td>0.0726367</td>
</tr>
<tr>
<td>c-a</td>
<td>-0.124900000</td>
<td>-0.19761688</td>
<td>-0.0621831171</td>
<td>0.0009694</td>
</tr>
<tr>
<td>d-a</td>
<td>-0.121033333</td>
<td>-0.18375022</td>
<td>-0.0081464505</td>
<td>0.0011961</td>
</tr>
<tr>
<td>c-b</td>
<td>-0.067366667</td>
<td>-0.13008355</td>
<td>-0.0046497838</td>
<td>0.0538549</td>
</tr>
<tr>
<td>d-b</td>
<td>-0.063500000</td>
<td>-0.12621688</td>
<td>-0.0007831171</td>
<td>0.0472664</td>
</tr>
<tr>
<td>d-c</td>
<td>0.003866667</td>
<td>-0.05885022</td>
<td>0.0665835495</td>
<td>0.9970454</td>
</tr>
</tbody>
</table>
> Rmax= aov(Rmax~Group, data=datal)
> summary(Rmax)

            Df  Sum Sq Mean Sq F value  Pr(>F)
Group        3 9.9765  3.3255 215.20 5.49e-08 ***
Residuals    8  0.0121  0.0015
---
Signif. codes: 0 *** 0.001 ** 0.01 * 0.05 . 0.1   1

> TukeyHSD(Rmax)
Tukey multiple comparisons of means
95% family-wise confidence level

Fit: aov(formula = Rmax ~ Group, data = datal)

$Group
diff   lwr      upr    p adj
b-a -0.349066667 -0.45075198 -0.2473814 0.0000195
 c-a -0.690466667 -0.79215198 -0.5887814 0.0000001
d-a -0.687340000 -0.78902531 -0.5856547 0.0000001
c-b -0.341400000 -0.44308531 -0.2397147 0.0000230
d-b -0.338273333 -0.43995864 -0.2365880 0.0000247
d-c  0.003126667 -0.09855864  0.1048120 0.9996279

> IP= aov(IP~Group, data=datal)
> summary(IP)

            Df  Sum Sq Mean Sq F value  Pr(>F)
Group        3 60.600   20.200 1539.22 2.20e-11 ***
Residuals    8  0.113    0.013
---
Signif. codes: 0 *** 0.001 ** 0.01 * 0.05 . 0.1   1

> TukeyHSD(IP)
Tukey multiple comparisons of means
95% family-wise confidence level

Fit: aov(formula = IP ~ Group, data = datal)

$Group
diff   lwr  upr    p adj
b-a 9.444333e-01 0.6448562 1.2440105 3.69e-05
c-a 4.916767e+00 4.6171895 5.2163438 0.00e+00
d-a 4.916767e+00 4.6171895 5.2163438 0.00e+00
c-b 3.972333e+00 3.6727562 4.2719105 0.00e+00
d-b 3.972333e+00 3.6727562 4.2719105 0.00e+00
d-c 8.881784e-16 -0.2995771 0.2995771 1.00e+00
3. Two-way ANOVA for temperature effects and their interactions

```r
> datam=read.csv(file.choose(),header=T)
> show(datam)

     Tb Ti  FR1  FR2  FR5  Rmax IP
1   80 120 1.0000 0.4830 0.1416 0.77460 0.0500
2   80 120 0.5100 0.4419 0.1213 0.63460 0.1167
3   80 120 0.9300 0.3442 0.1131 0.72170 0.0830
4   60 120 0.0450 0.1819 0.0860 0.32770 0.8500
5   60 120 0.0140 0.1574 0.1014 0.36370 1.2030
6   60 120 0.0047 0.1984 0.0160 0.37230 0.9500
7   80 100 0.0061 0.0120 0.0027 0.03090 5.0000
8   80 100 0.0220 0.0110 0.0023 0.02010 5.0000
9   80 100 0.0067 0.0002 0.0037 0.00850 5.0000
10  60 100 0.0160 0.0059 0.0034 0.01900 5.0000
11  60 100 0.0051 0.0035 0.0038 0.01869 5.0000
12  60 100 0.0055 0.0038 0.0057 0.03119 5.0000
> FR1Two=aov(FR1~Tb+Ti,data=datam)
> summary(FR1Two)

       Df Sum Sq Mean Sq F value Pr(>F)
Tb      1  0.6174  0.6174  8.91  0.0157 *
Ti      1  0.6097  0.6097  8.70  0.0162 *
Residuals 9  0.6307  0.0701
---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1

> FR2Two=aov(FR2~Tb+Ti,data=datam)
> summary(FR2Two)

       Df Sum Sq Mean Sq F value Pr(>F)
Tb      1  0.04581  0.04581  7.568  0.02241 *
Ti      1  0.26119  0.26119 43.153  0.000103 ***
Residuals 9  0.05448  0.00605
---
```
> FR2Two <- aov(FR2 ~ Tb + Ti, data = datam)
> summary(FR2Two)

    Df Sum Sq Mean Sq  F value Pr(>F)
Tb    1  0.0458  0.0458   7.568   0.0224 *
Ti    1  0.2612  0.2612  43.153  0.000103 ***
Residuals 9  0.0545  0.00605

---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 . ‘ 1

> FR2iTwo <- aov(FR2 ~ Tb + Ti + Tb:Ti, data = datam)
> summary(FR2iTwo)

    Df Sum Sq  Mean Sq  F value Pr(>F)
Tb    1  0.0458  0.0458   32.99   0.000432 ***
Ti    1  0.2612  0.2612  188.13 7.7e-07 ***
Tb:Ti 1  0.0433  0.0433  31.24  0.000517 ***
Residuals 8  0.0111  0.00139

---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 . ‘ 1

> FR5Two <- aov(FR5 ~ Tb + Ti, data = datam)
> summary(FR5Two)

    Df Sum Sq Mean Sq   F value Pr(>F)
Tb    1  0.0021  0.0021  2.616   0.1402
Ti    1  0.0266  0.0266 32.245  0.000302 ***
Residuals 9  0.0074  0.00082

---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 . ‘ 1

> FR5iTwo <- aov(FR5 ~ Tb + Ti + Tb:Ti, data = datam)
> summary(FR5iTwo)

    Df Sum Sq  Mean Sq  F value Pr(>F)
Tb    1  0.0021  0.0021  3.754  0.088668 .
Ti    1  0.0266  0.0266 46.270  0.000138 ***
Tb:Ti 1  0.0028  0.0028  4.914  0.057463 .
Residuals 8  0.0046  0.000575

---
> RmaxTwo <- aov(Rmax ~ Tb + Ti, data = datam)
> summary(RmaxTwo)

    Df Sum Sq Mean Sq F value   Pr(>F)
Tb     1 0.0898  0.0898  7.684  0.0217 *
Ti     1 0.7937  0.7937 67.950 1.74e-05 ***
Residuals 9 0.1051  0.0117
---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1

> RmaxiTwo <- aov(Rmax ~ Tb + Ti + Tb:Ti, data = datam)
> summary(RmaxiTwo)

    Df Sum Sq Mean Sq  F value   Pr(>F)
Tb     1 0.0898  0.0898  59.35  5.72e-05 ***
Ti     1 0.7937  0.7937 524.81 1.40e-08 ***
Tb:Ti  1 0.0930  0.0930  61.51  5.04e-05 ***
Residuals  8 0.0121  0.0015
---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1

> IPTwo <- aov(IP ~ Tb + Ti, data = datam)
> summary(IPTwo)

    Df Sum Sq Mean Sq  F value   Pr(>F)
Tb     1 0.67  0.6700  7.779  0.0211 *
Ti     1 59.26 59.2600 689.109 8.16e-10 ***
Residuals  9 0.77  0.0833
---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1

> IPiTwo <- aov(IP ~ Tb + Ti + Tb:Ti, data = datam)
> summary(IPiTwo)

    Df Sum Sq Mean Sq F value   Pr(>F)
Tb     1 0.67  0.6700  50.96  9.82e-05 ***
Ti     1 59.26 59.2600 4514.47 2.68e-12 ***
Tb:Ti  1 0.67  0.6700  50.96  9.82e-05 ***
Residuals  8 0.11  0.0138
4. Heat treatment effects on thin stillage

```r
> data1=read.csv(file.choose(),header=T)
> show(data1)

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Rf</th>
<th>Rf1</th>
<th>MaxR</th>
<th>IP</th>
<th>Ht</th>
<th>Cl</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.0710</td>
<td>0.3600</td>
<td>0.425</td>
<td>0.016</td>
<td>TS</td>
<td>1</td>
</tr>
<tr>
<td>2</td>
<td>0.0750</td>
<td>0.4920</td>
<td>0.435</td>
<td>0.000</td>
<td>E1</td>
<td>1</td>
</tr>
<tr>
<td>3</td>
<td>0.1000</td>
<td>0.9330</td>
<td>0.487</td>
<td>0.000</td>
<td>SK</td>
<td>1</td>
</tr>
<tr>
<td>4</td>
<td>0.1122</td>
<td>0.8270</td>
<td>0.537</td>
<td>0.000</td>
<td>E2</td>
<td>1</td>
</tr>
<tr>
<td>5</td>
<td>0.0610</td>
<td>0.0957</td>
<td>0.343</td>
<td>0.150</td>
<td>TS</td>
<td>1</td>
</tr>
<tr>
<td>6</td>
<td>0.0586</td>
<td>0.4340</td>
<td>0.418</td>
<td>0.016</td>
<td>E1</td>
<td>1</td>
</tr>
<tr>
<td>7</td>
<td>0.0622</td>
<td>0.5330</td>
<td>0.450</td>
<td>0.000</td>
<td>SK</td>
<td>1</td>
</tr>
<tr>
<td>8</td>
<td>0.0700</td>
<td>0.0940</td>
<td>0.510</td>
<td>0.000</td>
<td>E2</td>
<td>1</td>
</tr>
</tbody>
</table>

> Rfaov=aov(Rf~Ht,data=data1)
> summary(Rfaov)

    Df Sum Sq Mean Sq F value  Pr(>F)
Ht     3 0.0008768 0.0002923  0.653 0.622
Residuals 4 0.0017893 0.0004473

> Rf1aov=aov(Rf1~Ht,data=data1)
> summary(Rf1aov)

    Df Sum Sq Mean Sq F value Pr(>F)
Ht     3 0.4789 0.15965 5.373 0.069 .
Residuals 4 0.1189 0.02971

---
Signif. codes: 0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ 1

> MaxRaoov=aov(MaxR~Ht,data=data1)
> summary(MaxRaoov)

    Df Sum Sq Mean Sq F value  Pr(>F)
Ht     3 0.021302 0.007101  6.235 0.0547 .
Residuals 4 0.004555 0.001139

---
Signif. codes: 0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ 1

> IPaoov=aov(IP~Ht,data=data1)
> summary(IPaoov)

    Df Sum Sq Mean Sq F value Pr(>F)
Ht     3 0.009765 0.003255  1.43 0.358
Residuals 4 0.009106 0.002276
```
```r
> FR1aov=aov(FR1~Ht,data=data1)
> summary(FR1aov)
    Df Sum Sq Mean Sq F value Pr(>F)
  Ht    3 0.08363  0.02788  1.491  0.345
Residuals  4 0.07477  0.01869
> FR2aov=aov(FR2~Ht,data=data1)
> summary(FR2aov)
    Df Sum Sq Mean Sq F value Pr(>F)
  Ht    3 0.001648  0.0005495  0.185  0.901
Residuals  4 0.011859  0.0029647
> |
```

```r
> data2=read.csv(file.choose(),header=T)
> show(data2)
    Experiment RF RF1 MaxR IP Ht Cl
  1       9  0.0180  0.0180  0.0900000  0.570  TS  2
  2      10  0.0310  0.0310  0.1400000  0.280  E1  2
  3      11  0.0430  0.0523  0.2200000  0.220  SK  2
  4      12  0.0580  0.0580  0.2600000  0.083  E2  2
  5      13  0.0324  0.0324  0.1570000  0.250  TS  2
  6      14  0.0848  0.0848  0.3770000  0.000  E1  2
  7      15  0.0342  0.0342  0.14788276  0.032  SK  2
  8      16  0.0438  0.0438  0.17896581  0.022  E2  2
  9      17  0.0040  0.0040  0.02002549  1.120  TS  2
 10      18  0.0093  0.0093  0.04492454  0.650  E1  2
 11      19  0.0199  0.0199  0.05852691  0.567  SK  2
 12      20  0.0318  0.0318  0.13914763  0.183  E2  2
> Rfaov=aov(Rf~Ht,data=data2)
> summary(Rfaov)
    Df Sum Sq Mean Sq F value Pr(>F)
  Ht    3 0.001319  0.0004395  0.821  0.518
Residuals  8 0.004283  0.0005354
> Rfaov=aov(Rf1~Ht,data=data2)
> summary(Rfaov)
    Df Sum Sq Mean Sq F value Pr(>F)
  Ht    3 0.001269  0.0004231  0.738  0.559
Residuals  8 0.004588  0.0005735
> MaxRaov=aov(MaxR~Ht,data=data2)
> summary(MaxRaov)
    Df Sum Sq Mean Sq F value Pr(>F)
  Ht    3 0.02090  0.006966  0.629  0.616
Residuals  8 0.08855  0.011069
> IPaov=aov(IP~Ht,data=data1)
> summary(IPaov)
    Df Sum Sq Mean Sq F value Pr(>F)
  Ht    3 0.009765  0.003255  1.43  0.358
Residuals  4 0.009106  0.002276
```
> FR1aov=aov(FR1~Ht,data=data2)
> summary(FR1aov)

    Df  Sum Sq Mean Sq  F value Pr(>F)
Ht     3 0.004932 0.001644   1.42   0.307
Residuals 8 0.009261 0.001158

> FR2aov=aov(FR2~Ht,data=data2)
> summary(FR2aov)

    Df  Sum Sq Mean Sq F value Pr(>F)
Ht     3 0.003201 0.001066   1.15   0.386
Residuals 8 0.007413 0.0009266
> data3=read.csv(file.choose(),header=T)
> show(data3)

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Rf</th>
<th>RF1</th>
<th>MaxR</th>
<th>IP</th>
<th>Ht</th>
<th>Cl</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1</td>
<td>0.0710</td>
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<td>0.42500000</td>
<td>0.016</td>
<td>TS</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>0.0750</td>
<td>0.4920</td>
<td>0.43500000</td>
<td>0.000</td>
<td>E1</td>
</tr>
<tr>
<td></td>
<td>3</td>
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<td>0.9330</td>
<td>0.48700000</td>
<td>0.000</td>
<td>SK</td>
</tr>
<tr>
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<td>4</td>
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<td>0.8270</td>
<td>0.53700000</td>
<td>0.000</td>
<td>E2</td>
</tr>
<tr>
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<td>5</td>
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<td>0.0957</td>
<td>0.34300000</td>
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<td>TS</td>
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<tr>
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<td>6</td>
<td>0.0596</td>
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<td>0.41800000</td>
<td>0.016</td>
<td>E1</td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>0.0622</td>
<td>0.5330</td>
<td>0.45000000</td>
<td>0.000</td>
<td>SK</td>
</tr>
<tr>
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<td>8</td>
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<td>0.3940</td>
<td>0.51000000</td>
<td>0.000</td>
<td>E2</td>
</tr>
<tr>
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<td>9</td>
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<td>0.0180</td>
<td>0.09000000</td>
<td>0.570</td>
<td>TS</td>
</tr>
<tr>
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<td>0.0310</td>
<td>0.14000000</td>
<td>0.280</td>
<td>E1</td>
</tr>
<tr>
<td></td>
<td>11</td>
<td>0.0430</td>
<td>0.0523</td>
<td>0.22000000</td>
<td>0.220</td>
<td>SK</td>
</tr>
<tr>
<td></td>
<td>12</td>
<td>0.0580</td>
<td>0.0580</td>
<td>0.26000000</td>
<td>0.083</td>
<td>E2</td>
</tr>
<tr>
<td></td>
<td>13</td>
<td>0.0324</td>
<td>0.0324</td>
<td>0.15700000</td>
<td>0.250</td>
<td>TS</td>
</tr>
<tr>
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<td>14</td>
<td>0.0848</td>
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<td>0.000</td>
<td>E1</td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>0.0342</td>
<td>0.0342</td>
<td>0.14788276</td>
<td>0.032</td>
<td>SK</td>
</tr>
<tr>
<td></td>
<td>16</td>
<td>0.0438</td>
<td>0.0438</td>
<td>0.17896581</td>
<td>0.022</td>
<td>E2</td>
</tr>
<tr>
<td></td>
<td>17</td>
<td>0.0040</td>
<td>0.0040</td>
<td>0.02002549</td>
<td>1.120</td>
<td>TS</td>
</tr>
<tr>
<td></td>
<td>18</td>
<td>0.0093</td>
<td>0.0093</td>
<td>0.04492454</td>
<td>0.650</td>
<td>E1</td>
</tr>
<tr>
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<td>19</td>
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<td>0.0119</td>
<td>0.05952691</td>
<td>0.567</td>
<td>SK</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>0.0018</td>
<td>0.0018</td>
<td>0.13914763</td>
<td>0.163</td>
<td>E2</td>
</tr>
</tbody>
</table>

> Rfaov=aov(Rf~Ht, data=data3)
> summary(Rfaov)

<table>
<thead>
<tr>
<th>Df</th>
<th>Sum Sq</th>
<th>Mean Sq</th>
<th>F value</th>
<th>Pr(&gt;F)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ht</td>
<td>3</td>
<td>0.001683</td>
<td>0.0005610</td>
<td>0.585</td>
</tr>
<tr>
<td>Residuals</td>
<td>16</td>
<td>0.015350</td>
<td>0.0009594</td>
<td></td>
</tr>
</tbody>
</table>

> Rflaov=aov(RF1~Ht, data=data3)
> summary(Rflaov)

<table>
<thead>
<tr>
<th>Df</th>
<th>Sum Sq</th>
<th>Mean Sq</th>
<th>F value</th>
<th>Pr(&gt;F)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ht</td>
<td>3</td>
<td>0.2103</td>
<td>0.07009</td>
<td>0.631</td>
</tr>
<tr>
<td>Residuals</td>
<td>16</td>
<td>1.7765</td>
<td>0.11103</td>
<td></td>
</tr>
</tbody>
</table>

> MaxRaov=aov(MaxR~Ht, data=data3)
> summary(MaxRaov)

<table>
<thead>
<tr>
<th>Df</th>
<th>Sum Sq</th>
<th>Mean Sq</th>
<th>F value</th>
<th>Pr(&gt;F)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ht</td>
<td>3</td>
<td>0.0358</td>
<td>0.01193</td>
<td>0.363</td>
</tr>
<tr>
<td>Residuals</td>
<td>16</td>
<td>0.5253</td>
<td>0.03283</td>
<td></td>
</tr>
</tbody>
</table>

> IPaov=aov(IP~Ht, data=data3)
> summary(IPaov)

<table>
<thead>
<tr>
<th>Df</th>
<th>Sum Sq</th>
<th>Mean Sq</th>
<th>F value</th>
<th>Pr(&gt;F)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ht</td>
<td>3</td>
<td>0.3519</td>
<td>0.11730</td>
<td>1.379</td>
</tr>
<tr>
<td>Residuals</td>
<td>16</td>
<td>1.3607</td>
<td>0.08504</td>
<td></td>
</tr>
</tbody>
</table>
> RfTwo=aov(Rf~Ht+Cl, data=data3)
> summary(RfTwo)

             Df Sum Sq Mean Sq F value Pr(>F)
Ht           3 0.001683 0.000561 1.278 0.317859
Cl           1 0.008765 0.008765 19.967 0.000451 ***
Residuals   15 0.006585 0.000439
---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1
> RfITwo=aov(Rf1~Ht+Cl, data=data3)
> summary(RfITwo)

             Df Sum Sq Mean Sq F value  Pr(>F)
Ht           3 0.2103  0.0701  2.672  0.0849 .
Cl           1 1.3831  1.3831 52.738 2.78e-06 ***
Residuals   15 0.3934  0.0262
---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1
> MaxRTwo=aov(MaxR~Ht+Cl, data=data3)
> summary(MaxRTwo)

             Df Sum Sq Mean Sq F value  Pr(>F)
Ht           3 0.0358  0.0119  1.798  0.191
Cl           1 0.4258  0.4258 64.176 8.47e-07 ***
Residuals   15 0.0995  0.0066
---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1
> IPtwo=aov(IP~Ht+Cl, data=data3)
> summary(IPtwo)

             Df Sum Sq Mean Sq F value  Pr(>F)
Ht           3 0.3519  0.1173  1.948  0.1653
Cl           1 0.4573  0.4573  7.593  0.0147 *
Residuals   15 0.9034  0.0602
---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1

> FR1aov2=aov(FR1~Ht+Cl, data=data3)
> summary(FR1aov2)

             Df Sum Sq Mean Sq F value  Pr(>F)
Ht           1 0.0249  0.0249  2.873  0.108
Cl           1 0.6891  0.6891 79.344 8.2e-08 ***
Residuals   17 0.1476  0.0087
---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1
> FR2aov2=aov(FR1~Ht+Cl, data=data3)
> summary(FR2aov2)

             Df Sum Sq Mean Sq F value  Pr(>F)
Ht           1 0.0249  0.0249  2.873  0.108
Cl           1 0.6891  0.6891 79.344 8.2e-08 ***
Residuals   17 0.1476  0.0087
---
Signif. codes:  0 ‘***’ 0.001 ‘**’ 0.01 ‘*’ 0.05 ‘.’ 0.1 ‘ ’ 1