EFFECT OF DIFFERENT DIFFUSION STRATEGIES ON EXTRACTION OF SUGAR FROM SWEET SORGHUM

By

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EFFECT OF DIFFERENT DIFFUSION STRATEGIES ON SUGAR EXTRACTION FROM SWEET SORGHUM

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Abstract: Many studies have been reported suggesting sweet sorghum as an attractive feedstock for production of bio fuels. Sugar from sweet sorghum is generally extracted using a roller press but roller mills require high capital and operational cost and hence may not be economically viable for small scale industries. Diffusion processes could be used for multiple feedstocks, and may be an alternative method of sugar extraction for versatile small scale processes with multiple feedstocks.

Several diffusion techniques for extraction of free sugar from sweet sorghum were studied. Batch diffusion, fed batch diffusion, and counter current diffusion were evaluated for sugar extraction efficiencies from sweet sorghum. The effects of different parameters such as liquid to solid ratio (1.5, 3 and 4.5), contact time between solids and liquid (10 minutes, 15 minutes and 45 minutes) and temperature (60˚ C and 70˚C) during the diffusion process were evaluated. The effect of shaking (120 r.p.m) on batch and fed batch diffusion processes was also studied.

Results showed that liquid to solid ratio and temperature did not significantly impact sugar extraction during batch diffusion. Longer contact time between solids and liquid and shaking yielded significantly higher sugar during batch diffusion. Liquid to solid ratio, time, and temperature significantly affected fed batch and counter current diffusion processes. The highest yield of sugar, 70.81% maximum theoretical yield (MTY) was obtained at 70˚ C, 45 minute contact time and liquid to solid ratio of 3 during fed batch diffusion with shaking.

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CHAPTER I

INTRODUCTION

1.1 Background

Increasing energy consumption and depleting fossil fuel has created an urge to improve energy production (Rooney et al., 2007). By the end of 2025, the world's energy consumption is expected to rise by 57 percent compared to 2002, resulting in a search for alternative fuel resources to meet the rising energy demand (Office of the Biomass Program, Multi Year Program Plan 2007-2012). Moreover, consumption of fossil fuels is directly linked to carbon emissions and hence there is a desire to develop biofuels that generate low net carbon emissions (Rooney et al., 2007). The United States has the capability to produce 1.3 billion tons of dry biomass annually according to the United States Department of Agriculture (Perlack et al., 2005).

Biomass includes organic waste, animal waste, waste water, energy crops, and agricultural and industrial residues (Antonopoulou et al., 2008). Today, biomass is used for 14% of the world's total energy consumption (International energy agency, 1998). Biomass can be converted to biofuels using many techniques such as direct combustion, gasification, pyrolysis and biological treatments and biological conversion had economic and environmental advantages compared to other technologies (Demirbas, 2004).

Bio-fuels can be broadly classified into bio diesel and bio ethanol and the main focus is on bio ethanol as it can be directly blended with natural fuel and used in automobiles (Hansen, 2004). Bio-ethanol can be produced by directly fermenting sugars or from polysaccharides such as starch and cellulose that can be converted to sugar (Mussatto et al., 2010).

Ethanol that is produced from sugar crops such as sugar cane and sugar beets accounts for nearly 40% of the total bio ethanol produced and the remaining 60% is produced from starch crops (Biofuels platform, 2010).

Recently sweet sorghum has been found to be a potential feedstock for ethanol production due to its high productivity, ability to adapt to various climatic conditions, high sugar content (14-17 %) and tolerance for drought.

Sweet sorghum is a crop close to sugarcane in respect to its sucrose accumulation, ability to grow quickly and also to store sugar in its stalks (Prasad et al., 2007). Sugar cane requires nearly 12 months to completely mature whereas sweet sorghum requires 3-4 months. Sugarcane yields about 2.5-4.8 tons of sugar per acre whereas sweet sorghum yields 2-3 tons per acre (Kim and Day, 2011).

Sugar has been conventionally extracted from sugar cane by milling, where the sugarcane is subjected to roller mills to extract the sugary juice. Recently, diffusion has been adapted to extract sugar from sugar cane, a method in which solid-liquid extraction is employed to extract the sugar (Modesto et al., 2009).

Similarly, sugar extraction from sweet sorghum has traditionally been done with roller mills, but diffusion of sugar is also an option. In a temperate climate with smaller scale processing systems, one advantage of using diffusion is that it could be applied to multiple feedstocks. A versatile process such as sugar diffusion could be used for both sweet sorghum and sugar beets in a dual feedstock process.

1.2 Objectives

The main objective of the present study was to evaluate various diffusion options (batch diffusion, counter current diffusion, and fed batch diffusion) for extraction of sugar from sweet sorghum and to evaluate parameters that may affect sugar extraction efficiency.

The specific objectives were to:

A. Evaluate the effect of liquid to solid ratio on sugar extraction efficiencies.

B. Evaluate the effect of temperature on various sugar extraction efficiencies.

C. Evaluate the effect of time on different diffusion methods.

D. Evaluate the effect of shaking on batch and fed batch diffusion methods.

E. Compare sugar extraction efficiencies during different diffusion processes.

CHAPTER II

LITERATURE REVIEW

2.1 Sweet Sorghum

Sweet sorghum [*Sorghum bicolor (L.)(Weaver et al., 1997) Moench*] belongs to the grass family and is a relative of sugarcane (Saccharum spp.). Sweet sorghum is a C4 crop and has high a concentration of soluble sugars (Wu et al., 2010). In tropical and subtropical climates sweet sorghum grows vigorously. It has high photosynthetic efficiency and is resistant to drought (Wang et al., 2009).

It is the only crop which can be used for sugar, syrup, alcohol, jaggery, fodder, fuel and paper (Schaffert, 1992). In the United States, sweet sorghum was first introduced in the year 1852. It was called “Northern Sugar Plant” by Isaac Hedges because of its high sugar content. Sweet sorghum can grow up to 14 feet tall and produce 20-50 tons per acre in favorable conditions.

The energy efficiency of producing biofuels from sweet sorghum has been estimated at 1:8 (energy input: energy output), compared to corn which has been estimated to have energy efficiency of 1:1.8. Sugar concentrations of sweet sorghum were estimated to be 14-21° Brix (Rains et al., 1990).Sweet sorghum has high dry mass accumulation rates on a daily basis (Wiesenborn et al., 1999).

The composition of sweet sorghum was reported to be 12.4 % cellulose, 10.2 % hemicellulose, 4.8 % lignin, 55% sucrose, 3.2 % glucose and 0.3 % ash, expressed as percentage of dry weight (Billa et al., 1997).

Grains and stalks are the major components of sweet sorghum. Sorghum grain which is endosperm is the largest portion of the grain Endosperm of sorghum grain is the largest portion of the grain constituting about 80-85% of the grain that is made of starch- amylose and amylopectin. Sugars and starches are the main storage forms of energy. Cellulose and hemicellulose contribute to structural components in plants (Wall & Ross, 1970). Glucose and fructose are the two monosaccharaides and reducing sugars present in stem and leaf. Sucrose, a non-reducing sugar is the only major disaccharide (Reddy & Reddy, 2003).

Maltose is present in small amounts in the leaves and stems. Sweet sorghum contains 21% total sugars on a dry basis, out of which sucrose constitutes about 6-15%, glucose 0.5-5% and fructose 0-1.5%. The grain of the sorghum has about 5-6% sugars (Wall and Ross, 1970).

Reducing sugars are of highest concentration when sweet sorghum is about 40-45 days old. The center portion of the stalk is richest in sugars. The lower portion of the stalk contains more glucose than sucrose. Sugars in the leaf increase from 3-9% during the period of 2-3 weeks after bloom. A slight decrease in sugar occurs as starch is deposited in the grain. Starch is also present in the leaves and stem. Acid hydrolysable carbohydrates rise to 25% of dry weight shortly after the bloom (Wall and Ross, 1970).

Wall & Ross (1970) stated that in sweet sorghum stems, starch rises to 16 % but diminishes during grain formation. Hemicelluloses are present in the stem and leaf which are the major components of plant cell walls and other tissues. Cellulose in sweet sorghum is the major component of cell wall and is responsible for strength of the fibrous tissue.

Cellulose content doesn’t vary greatly during plant development. The stalk of the sweet sorghum can be divided into two parts: the pith fraction which contains most of the juice and sugars, and the rind section, which contains most of the fiber. The stalk rind is higher in cellulose than the pith. The expected yields of sweet sorghum are 42,000 kilograms of stalks per hectare, which yields approximately of 16,800 kilograms of juice (Wall & Ross, 1970).

For two decades, sweet sorghum has been considered an attractive feedstock for ethanol production. Its high water use efficiency, short growth period of 3-4 months and low fertilizer requirement make it an attractive biofuel feedstock (Rajvanshi, 2006).

2.2 Bioethanol

Bioethanol has received worldwide attention as a solution to global warming and an alternative fuel to gasoline (Hattori & Morita, 2010). Bioethanol, has many advantages as it is renewable, carbon neutral and the biomass feedstocks from which biofuels are produced are distributed everywhere, unlike petrol.(Kim and Dale, 2004) estimated that there is about 73.9 Tg of dry wasted crops in the world that could potentially produce 47.1 Giga Liters of ethanol.

Bioethanol production can be generally classified into three types based upon the type of raw material used:

- Bioethanol derived from sugar based material such as sugar cane, sugar beet, and sweet sorghum.
- Ethanol derived from starch based material such as grains of maize, sorghum, and wheat.
- Cellulosic bioethanol made from cellulosic materials such as agricultural residues, grasses, and trees.

USA and Brazil are the two leading countries in production of ethanol and have been using mainly maize and sugarcane. Nearly 18.3 billion liters of ethanol was produced by the United States ethanol industry in 2006 (Balat, 2009).

2.3 Other Feedstocks for Sugar Production

Raw materials that are used for sugar production to directly ferment to produce ethanol are sugar cane, sugar beet and sweet sorghum. Cane molasses was the only major raw material for ethanol production until recently. Molasses contains about 45-50% directly fermentable sugars which are available at a very low cost, making it an attractive raw material for biofuel production (Prasad et al., 2007).

2.3.1 Sugar Beets

Sugar beet roots contain high levels of sucrose and they are commercially grown for sugar. In 2011, United States, Germany, France and Russia were the world’s largest producers of sugar beets (Salzar et al., 2013). The root of the beet contains about 75% water, 20 % sugar and 5% pulp (Erdal et al., 2007). Sugar beets contain about 25% higher sugar compared to sugar cane (Cheesman, 2004).

Sugar beets grow in temperate zones, unlike sugarcane which grows in tropical and subtropical climates. The average weight of a sugar beet is 0.5-1 kilogram(Wall & Ross, 1970).

2.3.2 Sugar Cane

Sugarcane originated from India 2000 years ago. Cane sugar was leading the market until the sugar beet came into the market in the early nineteenth century. Sugar from sugar beets cannot be differentiated from sugar from sugarcane (Mitchell, 2004).

Sugar cane belongs to the grass family and the stalk of the cane grows up to 12 feet. The color of the stem varies from yellow to green to brownish red and the diameter of the stem varies from ¾ to 2 inches. A mature sugar cane stalk consists of 11-16 % fiber, 12-15% soluble sugars, 2-3% non-sugars and 63-73% water. The average yield of crop is 60-70 tons per hectare per annum. Sugarcane is a cash crop and is also used as livestock fodder in many parts of the world (Wall and Ross, 1970)

2.4 Sugar Processing Options

2.4.1 Diffusion

Diffusion is a sugar recovery technique using liquid extraction. The sugars are present in the tissue of the cane, which is not permeable and in order to make the material permeable, and diffuse the sugars through the cell wall, the temperature has to be increased. The cell wall colloids are precipitated, making solutes of comparatively low molecular weight free to move through the tissue towards the surrounding extraction liquid.

With the process of diffusion, high molecular weight substances such as sugars can be obtained without any impurities, as the cell wall acts as a molecular sieve. This is an advantage over mechanical expression of sugars, where the cells are broken and the whole contents are squeezed out. The concentration difference between the solute molecules within the cane and surrounding liquid is the driving force for the movement of solute molecules. No diffusion takes places when there is no concentration difference. Higher temperature will speed up the movement of solute particles from the tissue to the surrounding liquid. The larger the area of contact between the tissue and surrounding liquid the faster the concentration is equalized. Diffusion stops when the concentration gradient disappears, hence it is necessary that the concentration of sugar in the liquid is always at a minimum value for rapid diffusion to occur (Van der pol, 1986).

Rapid movement of this solution sometimes past the exposed tissue surface minimizes the possibility that a thin film of high concentration is built up in the immediate vicinity of the diffusion interface. Shortening the path of diffusion and increasing the diffusion interfacial area is simultaneously achieved by fine preparation of the cane and this preparation cannot be achieved by rupture of some juice cells which in turn allow high molecular non sugar components to escape into diffusion juice (Van der pol, 1986).

According to Fick's fundamental law of diffusion ds=k*(T/n).A*(dc/dx)*dt, the rate of diffusion is inversely proportional to square length of the path along which diffusion takes place, hence for maximum efficiency of diffusion the cane dimensions should be small and the regularity of the size must be maintained. Care should also be taken to minimize the cell rupture so that the unwanted high molecular weight particle doesn't mix with the sugar syrup (Van der pol, 1986).

Van der pol (1986) also stated that it is advantageous to place a single three roll mill before the diffuser, since 50 percent of the juice in the cane can be recovered. This would further make the feed stock more uniform for diffusion, which in turn increases diffusion efficiency. One additional advantage in placing the roller mill before diffusion is, that the load on the diffuser is reduced by nearly 50 percent. He also stated that, to obtain maximum concentration difference between the juices inside the cane tissue and that surrounding it, continuous counter current operation is the best system to use. Cane which was fully submerged in diffusing liquid all the time yielded higher sugars. He determined that when cane chip thickness was less 0.20 centimeters, 96% sugar extraction was achieved during diffusion at 180° F for 60 minutes. Van der pol expected nearly 98.9 percent overall extraction when a roller mill was used before diffusion of solids.

2.4.1.a Counter Current Diffusion

Multistage counter current extraction is a process that allows high recovery of solutes and produces an abstract with high solute concentration (Perry and Chilton, 1973). The process is generally carried out by moving the solids opposite to the solvent flow from stage to stage or accomplished by keeping the solids stationary in different sectors and pumping extracts with diminishing concentrations from one sector to another.

The counter current flow provides a greater overall driving force for mass transfer than co-current flow. Counter current extraction offers a high recovery of soluble solids and a high concentration of extract. Solute concentrations higher than equilibrium can be achieved using counter current extraction(Wiesenborn et al., 1999). Multi stage counter current extraction is widely used for extraction of coffee beans (Clarke, 1987).

Noah & Linden, (1989) discussed the effects of temperature and liquid to solid ration L/S on sugar and organic acid from ensiled sweet sorghum using a pilot scale continuous counter current diffuser. At unsteady state the pilot scale experimental diffuser was able to diffuse 90 % of the components of sweet sorghum. They used chopped sweet sorghum for countercurrent studies at temperatures of 50° C and 70° C and at five different liquid to solid ratios (2.5, 3.5, 4.0, 4.5 and 5.0). A liquid to solid ratio of 5 and a temperature of 70˚ C yielded the highest amount of sugar.

Yang & Brier (1958) performed continuous counter current diffusion of sugar beets. Results indicated that the liquid-film mass transfer coefficient of the beet-water system was independent of diffusion temperature and extraction liquid rate.

A multistage counter current extraction technique was developed for extraction of glycyrrhizic acid (GA). GA was extracted from *Glycyrrhiza uralensis* Fisch, a plant species of licorice. Many parameters such as extraction stage numbers, temperature, extraction time and solvent to licorice ratio on extraction yield of GA were tested. Optimum conditions were found to be five stages, 60 minutes extraction time for each stage, 60° C and 6 ml/g solvent to licorice ratio. Results from this study have indicated that multi stage counter current extraction offered the highest GA extraction yield compared to single pot extraction (SOP), microwave assisted extraction, ultrasonic extraction, Soxhlet extraction and room temperature extraction and saved time, energy and solvent consumption (Wang et al., 2004).

Rein & Woodburn (1974) performed diffusion experiments on sugar cane. A diffusion model was tested in two experimental configurations. The first model was a fixed bed pilot plant diffuser and the second one was a well-mixed pilot plant diffuser. Sugar diffusion was more efficient in the mixing environment.

Binkley & Wiley, (1978) studied the difference between continuous counter current diffusion and Willmes press (it is a batch mechanical press) on apple juice extraction. Results indicated that the counter current diffusion method yielded 13.4 % higher soluble solids.

2.4.1. b High Electric Field Pulses (HELP) Method of Diffusion

For more than one hundred years sugar extraction from sugar beets was done using thermal denaturation of sliced beets followed by diffusion in hot water at 70-75°C (Asadi, 2006). Thermal treatment leads to the breakage of cellular membrane and leads to tissue denaturation. The main objective of thermal denaturation is to improve transport of sugar through the tissue into the extracting liquid. The main disadvantage with this type of extraction is not that the membranes get destroyed but that cell walls also change their inner chemical structure through the reactions of hydrolytic degradation (Van der pol, 1986). Other cell components also penetrate the cell wall and pass into the juice; for example pectin penetrates the cell wall and deteriorates the quality of the juice (Van der pol, 1986). An alternative approach to avoid the contamination of cellular components is pulsed electric field (PEF). Under the PEF effect, the biological membrane becomes electrically pierced and loses its semi permeability and this loss may be temporary or permanent (Weaver et al., 1997). Loginova et al.(2011) performed PEF on sugar beets and could extract 17.8 to 20.8 g of sucrose per 100g of cossettes.

The application of HELP in food processing gained attention in the last decades utilizing its impact on cell membranes. Apart from food preservation, disintegration of biological tissue is often a key step in food processing prior to extraction of intercellular compounds. Applying HELP to cellular tissue, an increase in mass transfer coefficient was observed due to cell membrane permeabilization (Knorr et al., 1994).

HELP technology was first applied for disintegration of cells in Germany in the 1960's. It was reported that a 10-12% increase in juice yield was achieved when the HELP technique was used on apple tissue. The energy input required to achieve a disintegration of plant cells is in the range of 10-20 kJ/kg, and causes a minimum temperature increase of less than 5° C. The product quality will be retained in contrast to thermal treatments. Additionally, hot water extraction requires a significant amount of energy, as high as 175 kJ/kg of treated beet (Schultheiss et al., 2002).

A HELP treatment of sugar beets prior to extraction could allow the reduction of extraction temperatures or to apply mechanical pressing (Eshtiaghi & Yoswathana, 2012). The applicability of HELP has been investigated and results have shown that at a pulse number of 60, similar disintegration to a thermal treatment at 75° C for 15 min was obtained. A three step pressing at 5 Mpa pressure and addition of water intermediately was suggested to achieve high sugar content after a processing time of 30 min, in comparison to up to 90 min thermal extraction and the energy input required was 12 kJ/kg (Eshtiaghi & Knorr, 2002). Only one study reported the effect of high electric field pulses on sugar extraction from sugar cane using a 5kV/cm and 20 pulses for about 20 seconds and this yielded higher sugars compared to thermally treated sugarcane at 75° C for 15 minutes (Kuldiloke et al., 2008).

2.4.2 Mechanical Expression of Sugar Using Mills:

In this method, cane is directly fed to a series of roller mills, which mechanically squeeze out the juice from the cane. This method can be employed to both sweet sorghum and sugar cane. This is one of the oldest techniques of juice extraction from cane. Juice extraction rate depends upon mill speed, moisture content of the cane, the mill adjustment and feeding rate (Mask & Morris, 1991).

It has been shown that mechanically pressed raw juice has higher sugar concentration and contains less non sugars but juice yields remain unacceptable (Eshtiaghi & Yoswathana, 2012). With three-roller power mill, the juice extracted is about 50-60% of the weight of the cane. The yield of the juices is generally low in the case of sweet sorghum (Eshtiaghi & Yoswathana, 2012)

Gnansounou et al., (2005) converted sweet sorghum sugars to ethanol. They diffused sugars using sugar mill technology. It involved a series of tandem roller mills with counter current flow to leach the soluble sugars from sorghum. This yielded nearly 87 % of the sugars, which was calculated based on the proportion of initial sugars present in the juice after extraction from sweet sorghum. The sugar yield was 109 kg per ton of juice processed from sweet sorghum.

Tew et al., (2008) extracted juice from sweet sorghum using a core press method where sorghum was converted into chips and the chips were fed into a hydraulic press for 2 minutes. This method yielded 8-10% sugar.

Coble et al., (1984) tried three different diffusion techniques on sweet sorghum. In the first technique, they used a cage press with a pressure on shredded sweet sorghum of 10,000 kPa and covered the sorghum with sufficient water and heated it to 95° C for 5 minutes and it was allowed to cool to room temperature and then yeast was added for fermentation. The second study evaluated the effect of sorghum leaves on sugar and ethanol yield. Treatments were with stalks and leaves, leaf sheaths remaining on stalks and stalk only with sheath and leaves removed. The feed stock size was reduced using a hammer mill and diffused using water and then fermented. In the third, solids were removed from the mash before and after fermentation. Results were reported in terms of ethanol yield (200 proof) in liters per ton of sweet sorghum. Chopped sorghum yielded 45.5 liters of ethanol. Sweet sorghum with leaves yielded 37.2 l, stalks and leaf sheath yielded 38.5 l, mixture with stalks, leaves and sheath yielded 29.3 l of ethanol. By removing the solids before fermentation they could get about 29.3 l and after fermentation the yield was 31.5 l of ethanol.

Cundiff (1992) used a screw press to extract the sugar syrup from sweet sorghum, and the maximum yield was 52% of whole stalk sugar.Lamb et al. (1982) hand fed whole sorghum stalks to a typical roller press and could obtain an expression ratio of 0.58.

Broadhead (1972) chopped sweet sorghum (Rio variety) and expressed juice using a three roller mill and could obtain a sugar syrup of 19 ^{o} Brix.

Worley et al. (1992) estimated that a 30 kW screw press would require 567 MJ/ha to extract sugar syrup from sweet sorghum. Total fermentable sugar that was extracted using the screw press was 7600 kg/ha.

Gunasekaran et al., (1989) extracted soluble solids using a reversing, single screw counter current extractor from apple and pears. Solid concentrations of 12.3˚ Brix from apple and 10.6˚ Brix from pears was obtained

CHAPTER III

MATERIALS AND METHODS

3.1 Sweet Sorghum Feedstock

Sweet sorghum (variety M81E) was grown at the plant research facility on the campus of Oklahoma State University in Stillwater, OK. Stalks were hand harvested with machettes at the soft dough stage of maturity and processed immediately. Harvested stalks were chopped with a Seydelman bowl chopper, which is a shear mill used in the meat processing industry for creating meat emulsions. Whole sweet sorghum stalks were hand chopped to approximately 2-3 feet in length and fed directly into the bowl chopper to create particles of 2-4 cm in length. The chopped particles were stored in the freezer at -15°C to -20°C.

3.2 Moisture Content Analysis

Fine particles of sweet sorghum were oven dried at 105°C for 24 h and the difference in the initial and final weights was used to determine the moisture content of sweet sorghum.

3.3 Evaluation of Free Sugar Content

For evaluation of free sugar content, a protocol described by Maness and Sunkar (2010) was followed. Pre dried samples were finely ground prior to extraction of free sugars using a coffee grinder. Boiling ethanol (95 %) was used as the extraction solvent. Finely ground sorghum samples were placed in a 50 ml plastic centrifuge tube which was capped with a one hole rubber stopper with a reflux tube. This set up was boiled using a water bath at 85°C for 20 minutes. After 20 minutes the supernatant was centrifuged for 20 minutes at 10000 rpm and the supernatant was again separated from the centrifuge tube. This extraction was conducted 3 more times and the supernatant was collected and, was then filtered using a 0.45µ filter. The high performance liquid chromatography (HPLC) analysis of the filtered supernatant was performed using Agilent Technologies HPLC system with HPX-AENEX-87P column (Bio-Rad, Sunnyvale, CA, USA). D.I water was eluent at a flow rate of 0.6ml/min at 80°C. An RID detector was used.

3.4 Diffusion Tests Performed

3.4.1 Batch Diffusion

Finely chopped sweet sorghum particles were diffused for free sugars. For this type of diffusion process the sorghum particles were treated with hot water in conical flasks in triplicates. Sample size in each flask was 25 grams. The diffusion process was carried out in a hot water bath. After 10, 15, or 45 minutes of contact time, the sorghum particles were hand squeezed to separate the liquid portion using a strainer. The filtrate volume was measured and was used for further calculations. Parameters were chosen based on industrial methods, for all the diffusion processes that were tested in this study.

A completely randomized design was used to evaluate the following parameters during batch diffusion process:

1. Temperature [60°C and 70°C]
2. Time [10 minutes,15 minutes and 45 minutes]
3. Liquid to solid ratio [1.5, 3, and 4.5]

3.4.2 Counter Current Diffusion

To imitate the industrial counter current diffusion process where a conveyor belt is used for diffusion against water flowing in the opposite direction of the conveyor belt to maximize the mass transfer, a four stage counter current was set up in the lab using four flasks as shown in figure 3.1. For this set up 25 grams of sorghum solids were added to four different flasks labelled as A, B, C and D. Fresh water was first added to the flask A and after a designated contact time of 10/15/45 minutes, the liquid portion from flask A was transferred to flask B and fresh water was added to flask A. After the designated contact time, the liquid portion from flask B was separated and added to flask C and the liquid portion from A was added to B and fresh water was again added to flask A. After flask A received three fresh water treatments it was removed from the experiment. This procedure was continued until flasks B, C and D received one fresh water treatment each. This procedure was tested for different parameters such as liquid to solid ratio, temperature and contact time between liquid and solids. At the end of all fresh water treatments, the sugar solution from all the flasks was separated from the solids by hand squeezing. The final solution, which was a mixture of sugar solutions from A, B, C and D, was analyzed for sugar using the HPLC.

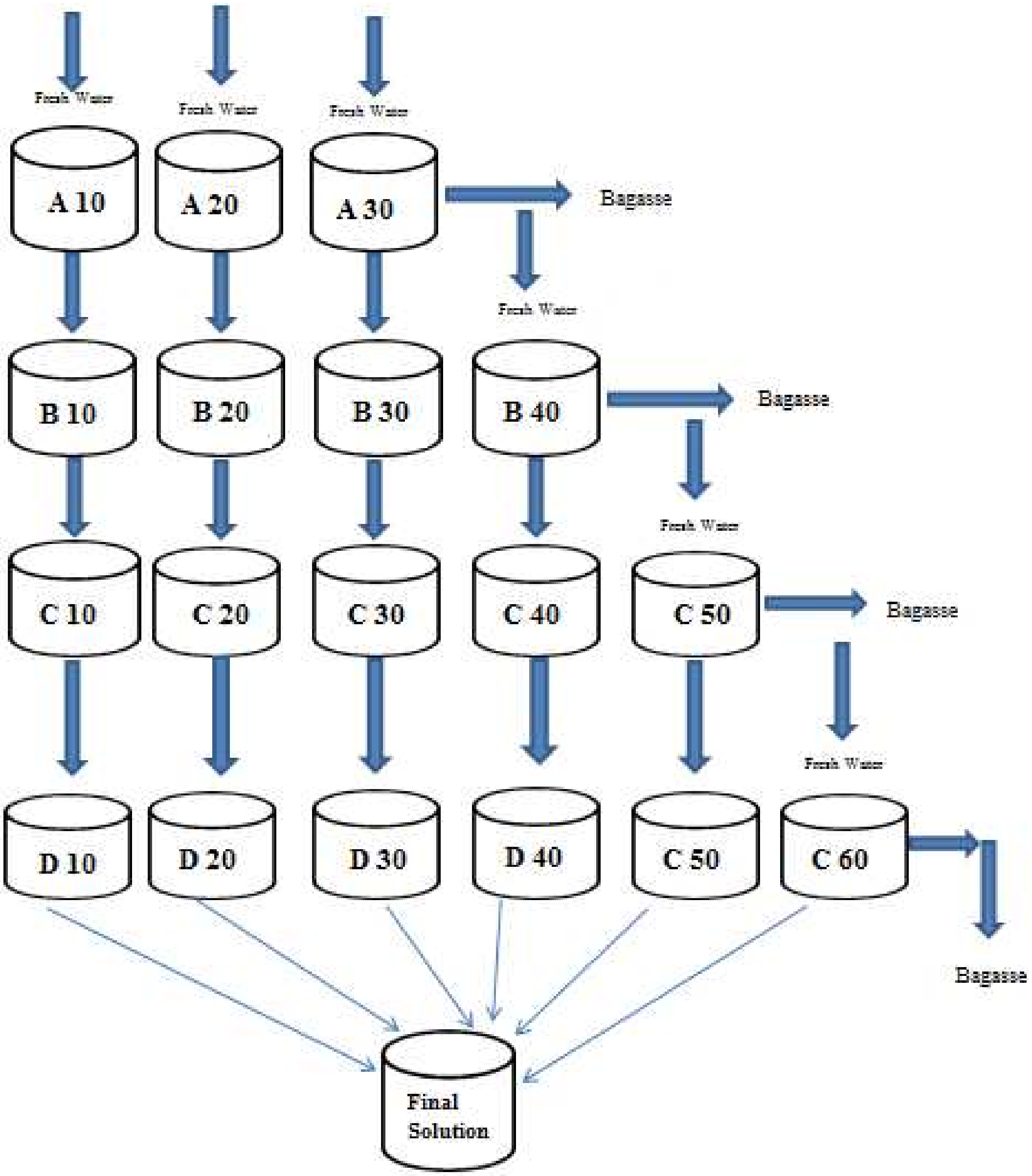


Fig. 3.1 Simulated counter current diffusion process with four sample flasks (A-D) and a 10 minute contact time.

A completely randomized design was used with the following variables during counter current diffusion:

1. Temperature [60, 70]
2. Time [10,15 and 45]
3. Liquid to solid ratio [1.5, 3,4.5]

3.4.3 Fed Batch Diffusion

During fed batch diffusion, sorghum particles were subjected to the same method as batch diffusion, but the contact time between the solids and liquid was divided into 3 steps instead of a single treatment. The liquid to solid ratio was divided into 3 steps along with the contact time. For example, a 3 step fed batch diffusion at 60° C, 15 minutes contact time and a liquid to solid ratio of 1.5 follows a procedure where L/S of 1.5 is split into 3 parts of 0.5 each and the contact time of 15 minutes is split into 3 parts of 5 minutes each. During the first step, L/S of 0.5 was used to extract sugar and the liquid portion was separated after 5 minutes, and same solids were diffused for another 5 minutes using another L/S of 0.5, and then the process was repeated a third time. Preliminary fed batch experiments using both 2 step and 3 step procedures showed that fed batch diffusion when carried out with 3 steps yielded 18 percent higher sugar than the 2 step procedure. Hence, all the experiments were carried out with a 3 step process and in a completely randomized design.

3.4.4 Batch and Fed Batch Diffusion with Shaking

For diffusion processes that included mixing, the same procedure was followed as that used for batch or fed batch diffusion. The only difference was that diffusion was carried out in a mechanical shaker operating at 120 rpm instead of a water bath.

3.5 Analytical Methods

All the sugar samples were centrifuged before analysis and the supernatant was filtered using a 0.45µ filter. The filtered samples were analyzed using high performance liquid chromatography (HPLC) analysis using Agilent Technologies HPLC system with HPX-AENEX-87P column (Bio-Rad, Sunnyvale, CA, USA). The elution was carried out using D.I water at a flow rate of 0.6ml/min at 80°C. An RID detector was used. The samples were analyzed for sucrose, glucose and fructose.

3.6 Calculations

Sucrose, glucose and fructose concentrations in gram per liter were obtained from HPLC results and a summation of all the sugars was used for total sugar concentration. The concentrations obtained were converted to amount of sugar in grams by multiplying it by the volume of sugar syrup obtained after each individual experiment. For example if 30 g/l was the concentration of the total sugars (sucrose, glucose and fructose) and 100 ml of sugar syrup was obtained after the experiment then 30 g/l * 0.1 liter which is 3 grams is the total amount of sugar obtained. Maximum Theoretical Yield (M.T.Y) was calculated using

$$\%\ \mathrm{M.T.Y} = \frac{\text{Sugar Diffused (grams)}}{\text{Intital Sugar in Sorghum}} * 100$$

3.7 Statistical Analysis

The effects of temperature, L/S ratio, contact time and shaking on total sugar extracted were analyzed in a mixed factorial design using the ADX tool in SAS (9.3) statistical software. Individual treatment effects were analyzed for significance using Duncan’s method. Significant difference was determined at α = 0.05.

CHAPTER IV

RESULTS AND DISCUSSION

4.1 Moisture and Soluble Sugar Content of Sweet Sorghum

The average moisture content of sweet sorghum after milling was found to be 75 ± 0.14%. The soluble sugar (sucrose + glucose + fructose) content was 3.28 grams for 25 grams of sweet sorghum, which is 13.13 ± 0.95 % on a wet basis. This was an average of 4 samples. The Percentage Maximum Theoretical Yield of Sugar was calculated based on this initial sugar content.

4.2 Batch Diffusion Process

The batch diffusion process was conducted with varying levels of liquid to solid ratio (3 levels: 1.5, 3 and 4.5), temperature (2 levels: 60° C and 70° C) and contact time (3 levels: 10 minutes, 15 minutes and 45 minutes).

4.2.1 Effect of Liquid to Solid Ratio

Figure 4.1 shows the maximum theoretical yield of sugar obtained during batch diffusion at various liquid to solid ratios and three different contact times at 70°C. From the figure it can be seen that the highest sugar yield of 36.98% was obtained at L/S 4.5 and the least was at L/S 1.5, which was 31.07% at the 10 minute contact time. With a 15 minute contact time, there was a 5% increase in sugar diffusion when L/S was increased from 1.5 to 3. Maximum sugar was diffused at L/S of 4.5 and 45 minute contact time, which was 49.22 %.

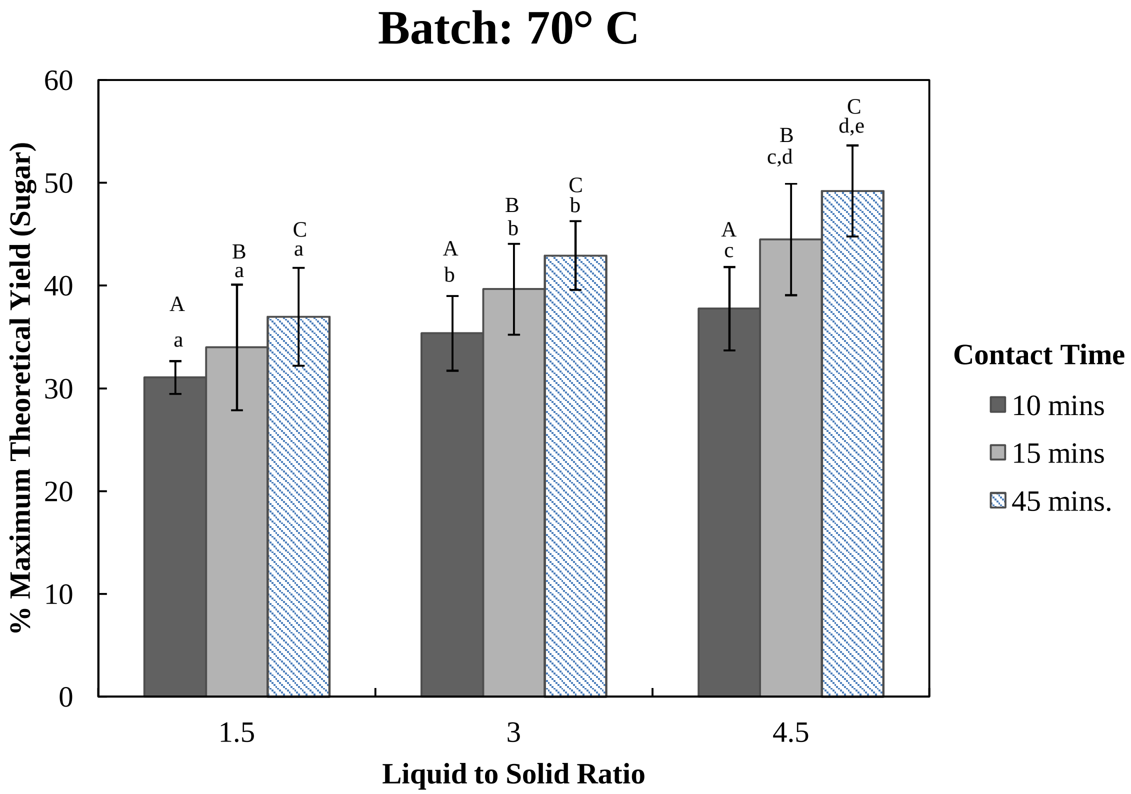


Fig. 4.1 Amount of free sugar diffused expressed as percentage of maximum theoretical yield at different liquid to solid ratios tested at 70° C and different contact times (10 minutes, 15 minutes and 45 minutes) during batch diffusion (n=6, error bars indicate standard error). Values at a given L/S that are followed by different lower case letters have significantly different sugar yields at different contact times. Values at a given contact time that are followed by different uppercase letters are significantly different sugar yields at different L/S. Significant difference is determined at α = 0.05.

Figure 4.2 shows the sugar yield obtained during batch diffusion at various liquid to solid ratios at 60° C. It can be seen that with a10 minute diffusion time, nearly the same amount of sugar was extracted when L/S ratio was increased from 1.5 to 4.5. When the L/S was increased from 1.5 to 3, the sugar diffusion increased by 7 %, but an increase in L/S from 3 to 4.5 did not show any change in sugar yield for the 15 minute diffusion time. The greatest amount of sugar, which was 48% of maximum theoretical yield, was obtained at L/S of 4.5 for the 45 minute contact time at 60°C.

ANOVA testing showed that the differences in sugar extraction due to the effect of liquid to solid ratio were not significantly different ($p > 0.05$). There are some clear trends suggesting that as the liquid to solid ratio increased, there was an increase in the amount of sugar extracted. This may be because of the increased contact area between water and sorghum solids. Maximum mass transfer can be achieved only when the contact area is large and hence more sugars may be diffused at higher L/S.

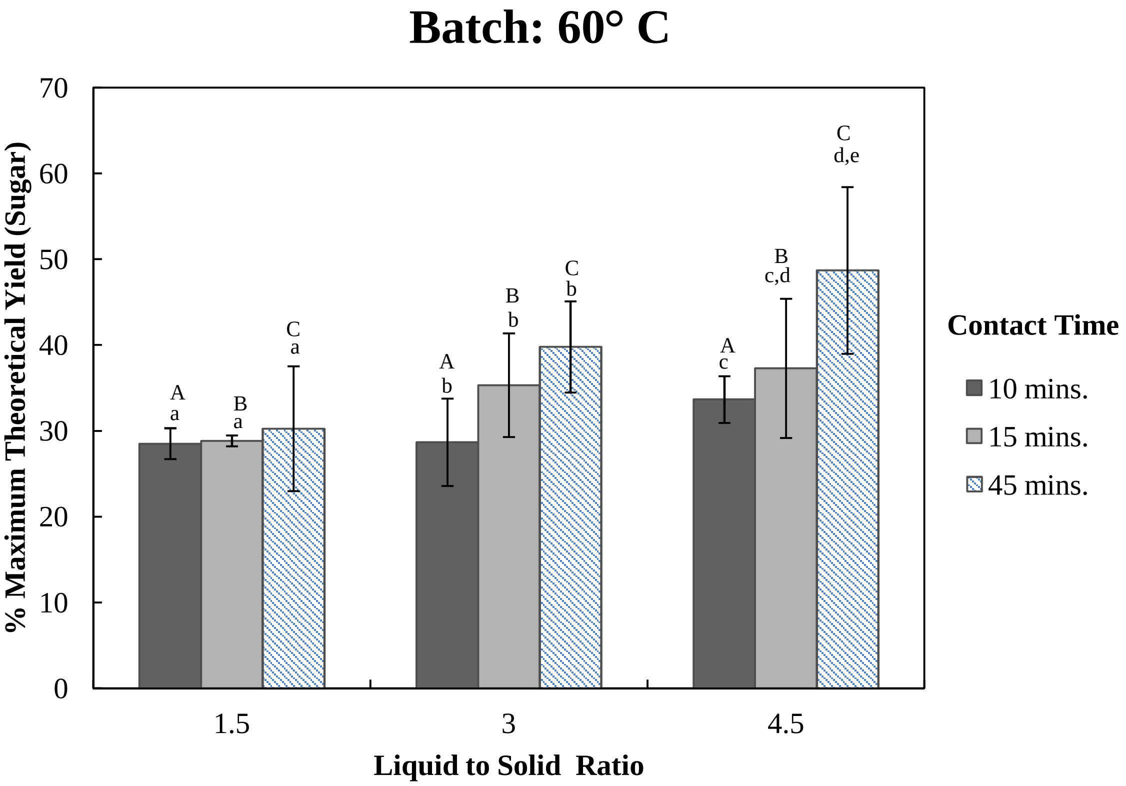


Fig. 4.2 Amount of free sugar diffused expressed as percentage of maximum theoretical yield at different liquid to solid ratios tested at 60° C and different contact times (10 minutes, 15 minutes and 45 minutes) during batch diffusion (n=6, error bars indicate standard error). Values at a given L/S that are followed by different lower case letters have significantly different sugar yields at different contact times. Values at a given contact time that are followed by different uppercase letters are significantly different sugar yields at different L/S. Significant difference is determined at α = 0.05.

4.2.2 Effect of Contact Time

Figures 4.1 and 4.2 show that the 45 minute contact time resulted in higher sugar yield at all liquid to solid ratios and both temperatures that were tested. Adequate contact time allowed maximum mass transfer from sorghum solids to the liquid around it. Shorter contact time was not sufficient to extract maximum sugars and hence 45 minute contact time yielded higher sugar. A statistical analysis showed that the contact time between liquid and solids had a significant effect on batch diffusion ($p < 0.0.5$). The amount of sugar diffused from sweet sorghum was significantly higher with 45 minutes contact time compared to 10 minutes contact time at a L/S of 4.5 and at temperatures of 60^o C and 70^o C.

4.2.3 Effect of Temperature

Table 4.1 compares the sugar yields at both temperatures (60^o C and 70^o C) at all levels of liquid to solid ratio and all three contact times. From the table it can be seen that 70^o C yielded higher sugar compared to 60^o C at all contact times and liquid to solid ratios. Intuitively, this makes sense because as the temperature increases, the kinetic energy of the molecules increases, which helps in faster movement of sugar from sorghum solids to the solution around it and thereby increases the mass transfer rate. Although there is a clear trend showing increased sugar yield with increased temperature, a statistical analysis revealed that there was no significant change in sugar yield when temperature was increased from 60^o C to 70^o C ($p > 0.05$).

TABLE 4.1

Effect of Temperature on Batch Diffusion. Average is calculated using 6 reps. Significant difference is determined at α = 0.05.

Liquid to Solid Ratio	Contact Time (minutes)	Temp.(°C)	Sugar (g) Avg. ± Std. Error	% Max. Theoretical Yield Sugar	Significance Yes (Y) or No (N)
1.5	10	60	0.93 ± 0.06	28.51	N
1.5	10	70	1.02 ± 0.11	31.07	
1.5	15	60	0.94 ± 0.06	28.84	N
1.5	15	70	1.11 ± 0.11	33.99	
1.5	45	60	0.99 ± 0.75	30.25	N
1.5	45	70	1.21 ± 0.21	36.98	
3.0	10	60	0.94 ± 0.02	28.66	N
3.0	10	70	1.16 ± 0.13	35.36	
3.0	15	60	1.16 ± 0.07	35.33	N
3.0	15	70	1.30 ± 0.10	39.66	
3.0	45	60	1.30 ± 0.31	39.78	N
3.0	45	70	1.41 ± 0.07	42.95	
4.5	10	60	1.10 ± 0.06	33.66	N
4.5	10	70	1.24 ± 0.14	37.77	
4.5	15	60	1.22 ± 0.14	37.28	N
4.5	15	70	1.46 ± 0.41	44.50	
4.5	45	60	1.59 ± 0.16	48.71	N
4.5	45	70	1.61 ± 0.13	49.22	

4.2.4 Effect of Shaking

Figure 4.3 shows the effect of shaking on sugar extraction during the batch diffusion process at different liquid to solid ratios at a temperature of 70^{o} C and contact time of 45 minutes. From the figure it can be seen that shaking greatly improved the sugar diffusion process. Sugar yields with shaking at liquid to solid ratios of 3 and 4.5 were 57.20% and 59.88%, respectively, with each one being about 10% higher than the similar treatment without shaking. ANOVA testing showed that sugar yields with shaking were significantly higher than those without shaking during batch diffusion ($P < 0.05$).

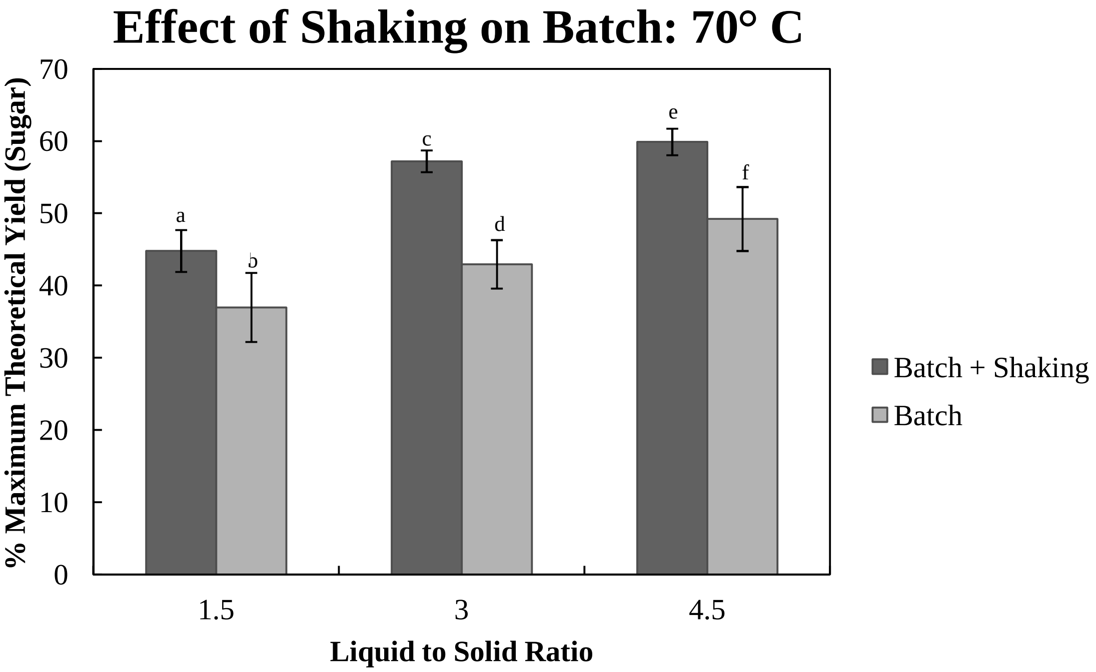
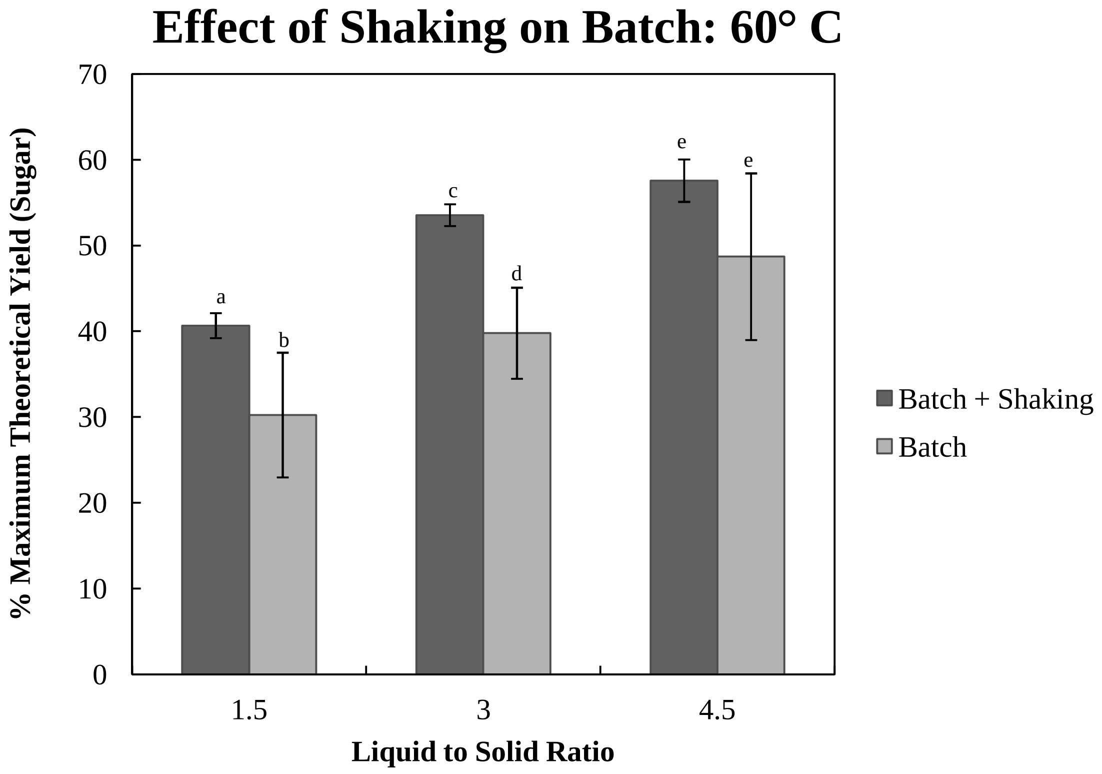


Fig. 4.3 Amount of free sugar diffused expressed as percentage of maximum theoretical yield at different liquid to solid ratios tested at 70° C and 45 minutes contact time during batch diffusion with and without shaking at 120 r.p.m (n=6, error bars indicate standard error). Values at a given L/S that are followed by different letters are significantly different. Significant difference is determined at $\alpha = 0.05$.

Figure 4.4 shows the effect of shaking at 60° C during the batch diffusion process. Again, there was a trend showing increased sugar diffusion with shaking. At all liquid to solid ratios, when the samples were shaken, sugar yields were about 10% higher than without shaking under the same conditions at 60° C. The effect of shaking was significant ($p < 0.05$) at liquid to solid ratios of 1.5 and 3.



.**Fig. 4.4** Amount of free sugar diffused expressed as percentage of maximum theoretical yield at different liquid to solid ratios tested at 60° C and 45 minutes contact time during batch diffusion with and without shaking at 120 r.p.m, (n=6, error bars indicate standard error). Values at a given L/S with different letters are significantly different. Significant difference is determined at α = 0.05.

4.3 Fed Batch Diffusion

In this type of diffusion, sorghum particles were subjected to same method as batch diffusion, the fresh water was added in 3 steps rather than all at one time, splitting but the contact time between the solids and liquid into 3 steps instead of a single treatment. The fed batch process was conducted with various levels of liquid to solid ratio (3 levels: 1.5, 3 and 4.5), temperature (2 levels: 60° C and 70° C) and time (3 levels: 10 minutes, 15 minutes and 45 minutes).

4.3.1 Effect of Liquid to Solid Ratio

Figure 4.5 shows the maximum theoretical sugar yield during fed batch diffusion at 70^o C and varying liquid to solid ratios for three different contact times. From the figure it can be seen that there is a clear trend showing increased sugar diffusion with increased liquid to solid ratio. At 10 minutes diffusion time, the highest yield of 42.97% MTY was obtained at L/S of 4.5 and the least was at L/S of 1.5, which was 30.27% MTY. For 15 minutes diffusion time, the amount of sugar extracted was up by nearly 10% when L/S was increased from 1.5 to 3. The maximum sugar was diffused at L/S of 4.5 and a 45 minute contact time, which was 56.22 %.

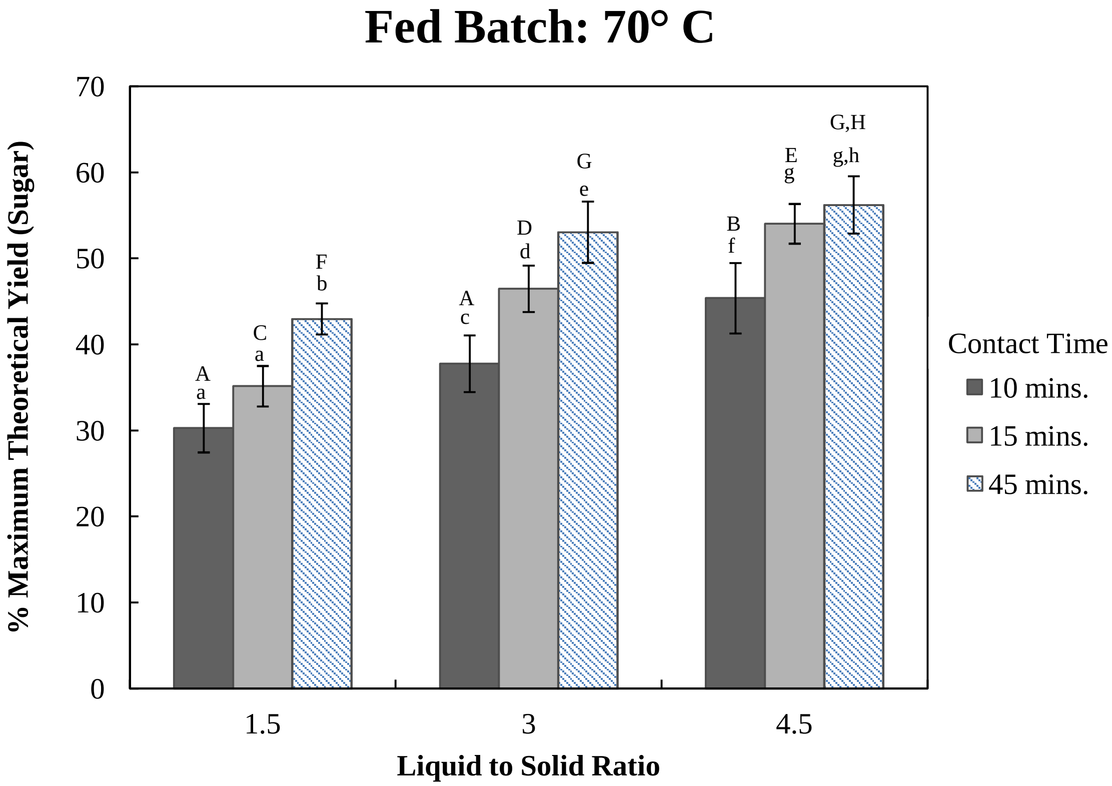


Fig. 4.5 Amount of free sugar diffused expressed as percentage of maximum theoretical yield at different liquid to solid ratios tested at 70°C and different contact times (10 minutes, 15 minutes and 45 minutes) during fed batch diffusion (n=6, error bars indicate standard error). Values at a given L/S that are followed by different lower case letters have significantly different sugar yields

at different contact times. Values at a given contact time that are followed by different uppercase letters are significantly different sugar yields at different L/S. Significant difference is determined at $\alpha = 0.05$.

Figure 4.6 shows the sugar yields at varying liquid to solid ratios during fed batch diffusion at 60^{o}C. The same trend showing increased sugar diffusion with increased liquid to solid ratio occurred at 60° C, but the differences were not that great. At 10 minutes diffusion time, there was not much change in the amount of diffused sugar when liquid to solid ratio was increased from 1.5 to 3 and 4.5. For the 45 minute diffusion time, at L/S of 4.5, the sugar extraction was 52.66%, which was 10 % and 21 % higher compared to L/S ratios of 3 and 1.5 respectively.

Although there was a trend showing increased sugar diffusion with increased liquid to solid ratio, statistical analysis results showed that the differences due to liquid to solid ratio were not statistically significant at 60^{o}C ($p > 0.05$). However, at 70^{o}C, a significant difference in sugar yield was observed at all liquid to solid ratios and contact times ($p < 0.05$) except for L/S of 1.5 and 3 with 10 minutes diffusion time and L/S of 3 and 4.5 with 45 minute contact time.

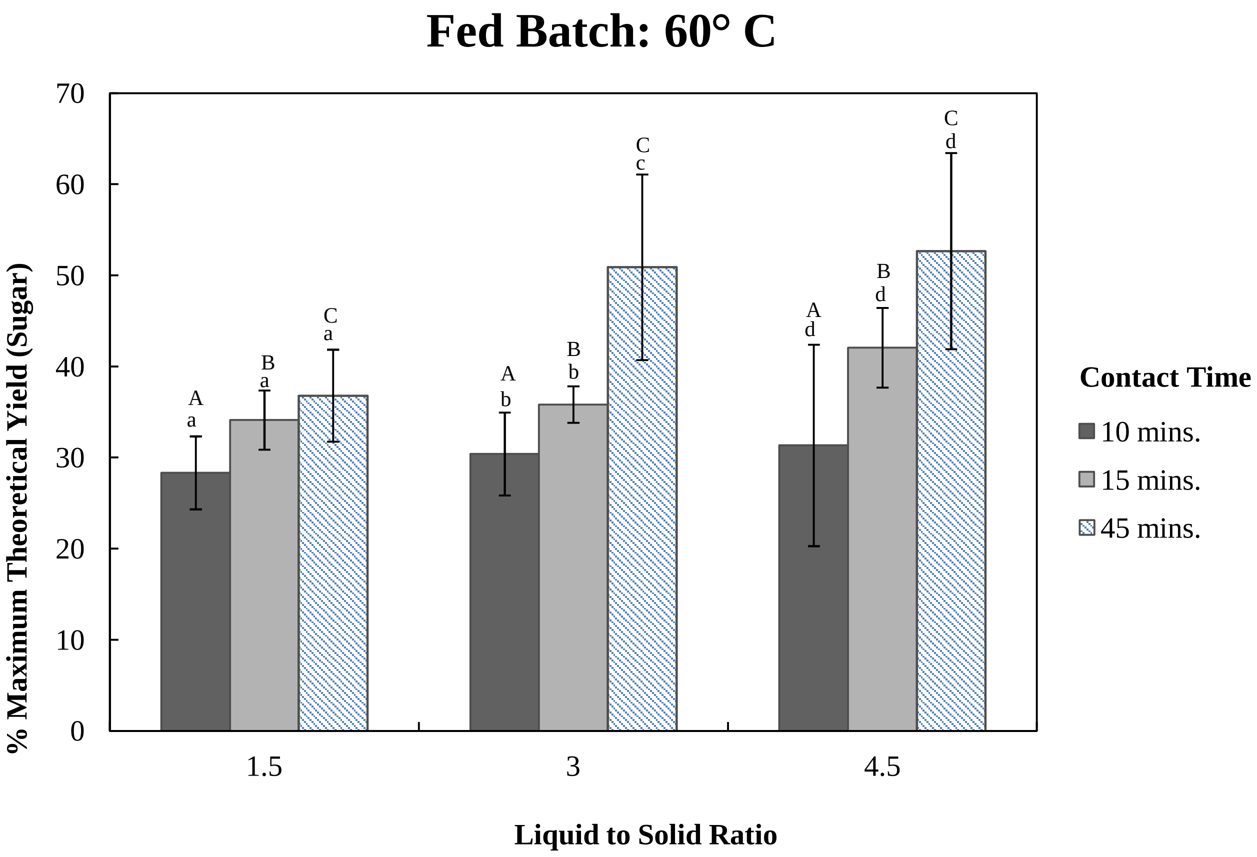


Fig. 4.6 Amount of free sugar diffused expressed as percentage of maximum theoretical yield at different liquid to solid ratios tested at 60°C and different contact times (10 minutes, 15 minutes and 45 minutes) during fed batch diffusion (n=6, error bars indicate standard error). Values at a given L/S that are followed by different lower case letters have significantly different sugar yields at different contact times. Values at a given contact time that are followed by different uppercase letters are significantly different sugar yields at different L/S. Significant difference is determined at α = 0.05.

4.3.2 Effect of Temperature

Table 4.2 shows a direct comparison of sugar yields at temperatures of 60^o C and 70^o C at all levels of liquid to solid ratio and contact time during fed batch diffusion. From the table it can be seen that 70° C yielded higher sugar compared to 60° C at all contact times and liquid to solid ratios. Again, this makes sense due to the increased movement of molecules at higher temperatures. Statistically, the temperature difference was not significant under most conditions, but there was a significant difference between the 60^{o} and 70^{o} diffusion temperatures at liquid to solid ratios of 3 and 4.5 with 15 minutes contact time ($P < 0.05$).

TABLE 4.2

Effect of Temperature on Fed Batch Diffusion. Average is calculated using 6 reps. Significant difference is determined at $\alpha = 0.05$.

Liquid to Solid Ratio	**Contact Time (minutes)**	**Temp.(°C)**	**Sugar (g) Avg. ± St. Error**	**% Max. Theoretical Yield Sugar**	**Significance Yes (Y) or No (N)**
1.5	10	60	0.93 ± 0.13	28.36	N
1.5	10	70	0.99 ± 0.11	30.28	
1.5	15	60	1.11 ± 0.09	33.89	N
1.5	15	70	1.15 ± 0.18	35.15	
1.5	45	60	1.20 ± 0.15	36.80	N
1.5	45	70	1.41 ± 0.07	42.98	
3.0	10	60	0.99 ± 0.11	30.40	N
3.0	10	70	1.24 ± 0.09	37.77	
3.0	15	60	1.17 ± 0.08	35.82	Y
3.0	15	70	1.52 ± 0.12	46.48	
3.0	45	60	1.67 ± 0.19	50.90	N
3.0	45	70	1.72 ± 0.09	52.55	
4.5	10	60	1.02 ± 0.33	31.29	N
4.5	10	70	1.49 ± 0.14	45.39	
4.5	15	60	1.38 ± 0.10	42.07	Y
4.5	15	70	1.77 ± 0.18	54.04	
4.5	45	60	1.57 ± 0.24	48.05	N
4.5	45	70	1.84 ± 0.11	56.23	

4.3.3 Effect of Contact Time

Figures 4.5 and 4.6 show that the longer 45 minute contact time resulted in higher sugar yields at all liquid to solid ratios and temperatures that were tested during fed batch diffusion. Adequate contact time allowed maximum mass transfer from sorghum solids to the liquid around it, and the shorter contact times were not as efficient. Significantly ($p < 0.05$) higher sugar was obtained at 45 minutes compared to the 10 minute and 15 minute contact times at 70˚ C and liquid to solid ratio of 1.5, and also at a liquid to solid ratio of 3 and temperatures of 60˚ C and 70˚ C.

4.3.4 Effect of Shaking

Figure 4.7 shows the effect of shaking on fed batch diffusion at 70^{o} C and several different liquid to solid ratios. From the figure it is clear that the addition of shaking to the fed batch diffusion process improves the sugar extraction efficiency. The average sugar yields are about 10% higher with shaking than without it. Statistical analysis showed that sugar yields were significantly higher with shaking than without shaking at all liquid to solid ratios.

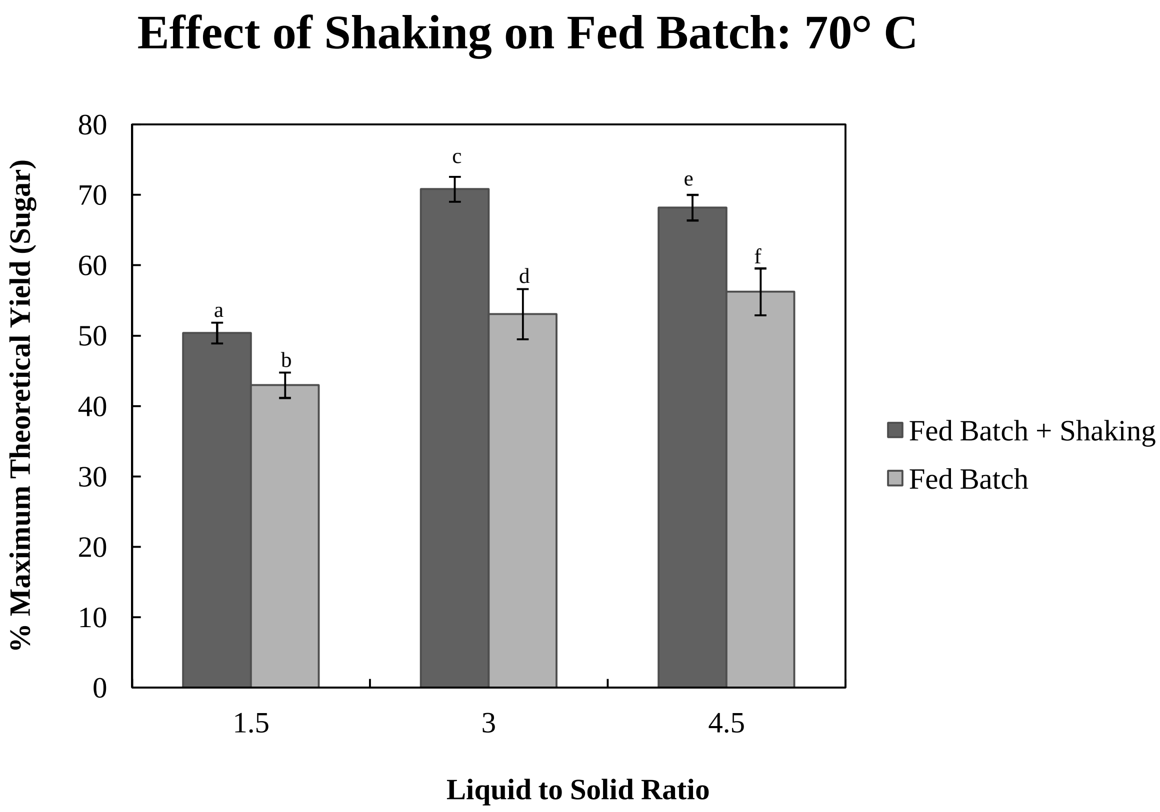


Fig. 4.7 Amount of free sugar diffused expressed as percentage of maximum theoretical yield at different liquid to solid ratios tested at 70°C and 45 minutes contact time during fed batch diffusion with and without shaking at 120 r.p.m (n=6, error bars indicate standard error). Values at a given L/S with different letters are significantly different. Significant difference is determined at α = 0.05.

Figure 4.8 shows the effect of shaking on fed batch diffusion at 60^{o} C. Similarly, it is clear that the addition of shaking to the fed batch diffusion process increases the sugar yield. At 60°C the sugar diffusion with shaking was significantly higher than that without shaking at both the L/S of 3 and 4.5, but was not significantly different at L/S of 1.5 during fed batch diffusion.

The positive effect of shaking on diffusion in the present study was similar to a previous study involving sugar cane where sugar diffusion was more efficient in a mixing environment. (Rein & Woodburn, 1974).

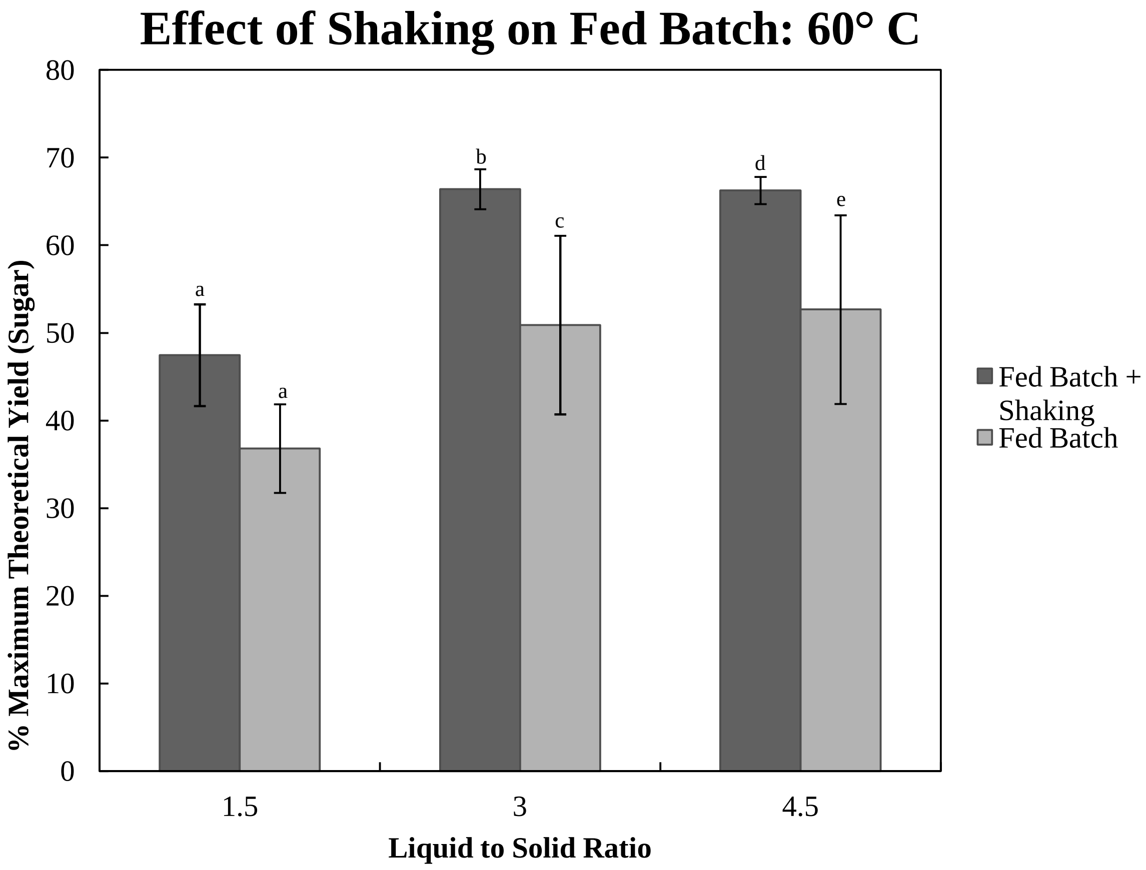


Fig. 4.8 Amount of free sugar diffused expressed as percentage of maximum theoretical yield at different liquid to solid ratios tested at 60° C, 45 minutes contact time during fed batch diffusion with and without shaking at 120 rpm (n=6, error bars indicate standard error). Significant difference is determined at α = 0.05.

4.4 Counter Current Diffusion Process

A four stage counter current diffusion process was set up in the lab using four flasks (A-D) to imitate an industrial counter current diffusion process. It was conducted with different levels of liquid to solid ratio (3 levels: 1.5, 3 and 4.5), temperature (2 levels: 60° C and 70° C) and time (3 levels: 10 minutes, 15 minutes and 45 minutes). Values at a given L/S with different letters are significantly different.

4.4.1 Effect of Liquid to Solid Ratio

Figure 4.9 shows the maximum theoretical yield of sugar obtained during counter current diffusion at various liquid to solid ratios and three different contact times at 70°C. From the figure it can be seen that the highest sugar yield of 49.04 % MTY was obtained at L/S 4.5 with a 45 minute contact time and the least was at L/S of 1.5 which was 35.63% MTY with a 10 minute contact time.

Statistical analysis revealed that L/S significantly ($P < 0.05$) affected sugar diffusion with a 15 minute contact time.

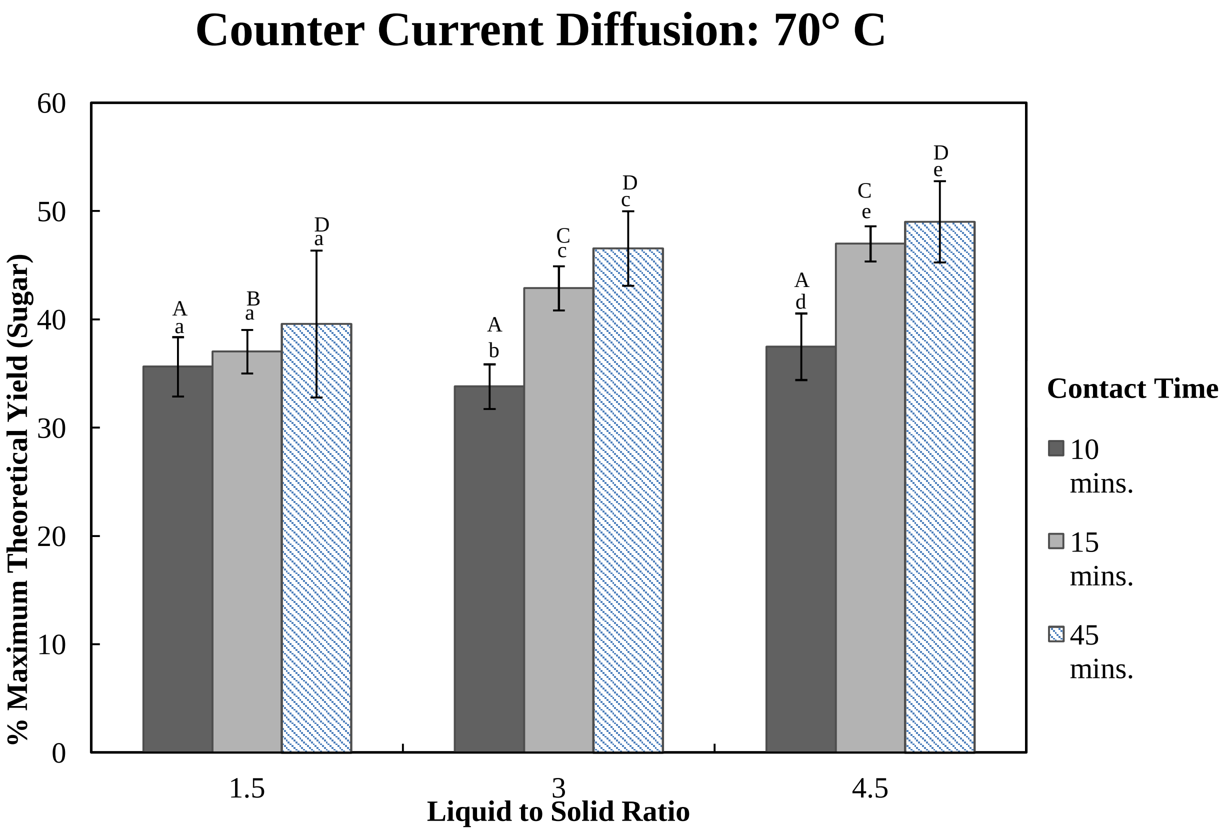


Fig. 4.9 Amount of free sugar diffused expressed as percentage of maximum theoretical yield Vs different liquid to solid ratios tested at 70°C and different contact times (10 minutes, 15 minutes and 45 minutes) during counter current diffusion (n=6, error bars indicate standard error). Values at a given L/S that are followed by different lower case letters have significantly different sugar yields at different contact times. Values at a given contact time that are followed by different uppercase letters are significantly different sugar yields at different L/S. Significant difference is determined at α = 0.05.

Figure 4.10 shows the sugar yields at varying liquid to sold ratios during counter current diffusion at 60° C. At contact times of 10 and 15 minutes, there is no clear trend in the sugar diffusion as affected by liquid to solid ratio. However, at a contact time of 45 minutes, significantly ($p < 0.05$) higher sugar was diffused at L/S of 4.5 with a contact time of 45 minutes compared to L/S of 1.5 and 3.

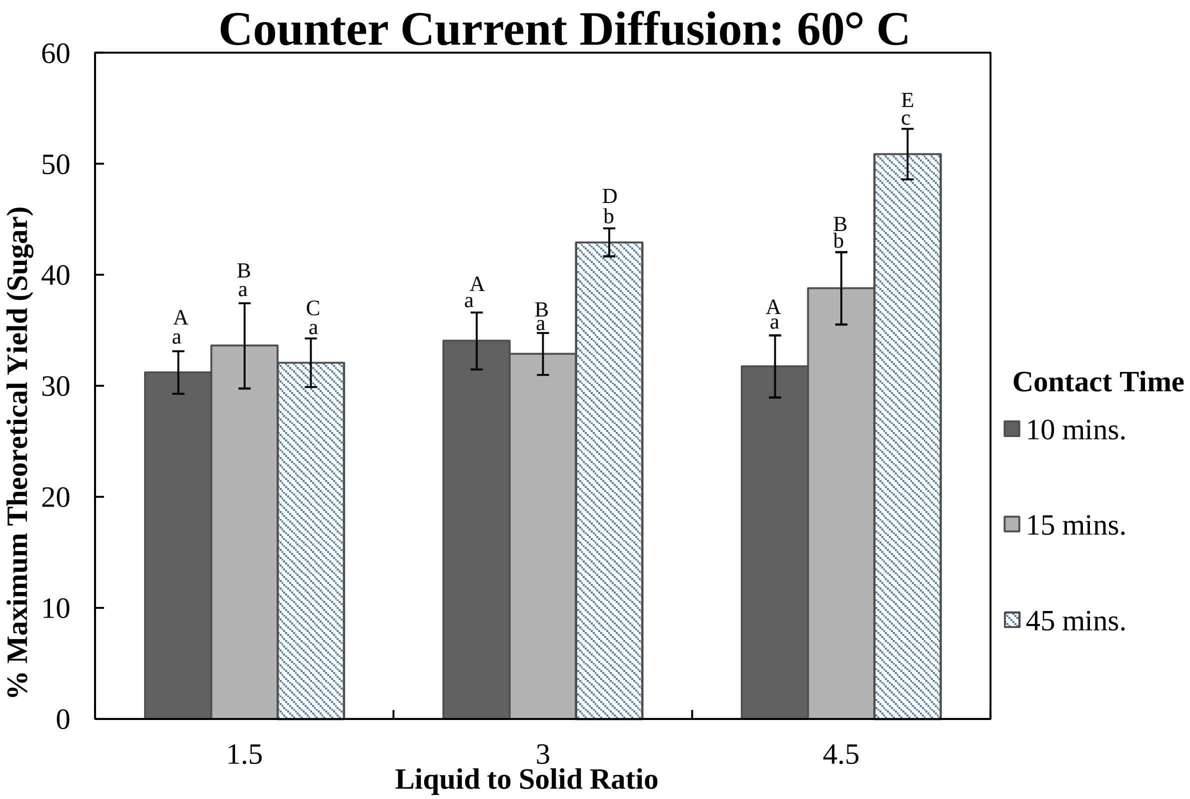


Fig. 4.10 Amount of free sugar diffused expressed as percentage of maximum theoretical yield at different liquid to solid ratios tested at 60°C and different contact times (10 minutes, 15 minutes and 45 minutes) during counter current diffusion (n=6, error bars indicate standard error). Values at a given L/S that are followed by different lower case letters have significantly different sugar yields at different contact times. Values at a given contact time that are followed by different uppercase letters are significantly different sugar yields at different L/S. Significant difference is determined at $\alpha = 0.05$.

4.4.2 Effect of Contact Time

Figures 4.9 and 4.10 show that the 45 minute contact time resulted in higher sugar yields than the 10 and 15 minute contact times at most liquid to solid ratios and both temperatures that were tested. Maximum mass transfer took place from sorghum solids to liquid with adequate contact time. A statistical analysis showed that the contact time between liquid and solids had a significant effect on counter current diffusion. Significantly ($P < 0.05$) higher sugar was diffused with 45 minute contact time compared to the 10 minute contact time at L/S of 3 and 4.5 at 70° C.

4.4.3 Effect of Temperature

Table 4.3 compares the sugar yields at both temperatures (60^o C and 70^o C) at all levels of liquid to solid ratio and all contact times during counter current diffusion. From the table it can be seen that 70° C generally yielded higher sugar compared to 60^o C. Statistical analysis showed that the amount of sugar diffused was significantly higher at 70^o C with contact times of 15 and 45 minutes at L/S of 3.

TABLE 4.3

Effect of Temperature on Counter Current Diffusion. Average is calculated using 6 reps. Significant difference is determined at α = 0.05.

Liquid to Solid Ratio	Time (minutes)	Temp.(°C)	Sugar (g) Avg. ± St. Error	% Max. Theoretical Yield Sugar	Significance Yes (Y) or No (N)
1.5	10	60	1.02 ± 0.06	31.21	N
1.5	10	70	1.17 ± 0.07	35.62	
1.5	15	60	1.10 ± 0.09	33.63	N
1.5	15	70	1.21 ± 0.09	37.02	
1.5	45	60	1.05 ± 0.08	32.09	N
1.5	45	70	1.30 ± 0.25	39.60	
3.0	10	60	1.11 ± 0.13	34.04	N
3.0	10	70	1.11 ± 0.07	33.81	
3.0	15	60	1.08 ± 0.07	32.90	Y
3.0	15	70	1.11 ± 0.11	33.81	
3.0	45	60	1.41 ± 0.06	42.95	Y
3.0	45	70	1.52 ± 0.13	48.49	
4.5	10	60	1.04 ± 0.07	31.76	N
4.5	10	70	1.23 ± 0.05	37.50	
4.5	15	60	1.27 ± 0.09	38.81	N
4.5	15	70	1.54 ± 0.07	47.01	
4.5	45	60	1.67 ± 0.04	50.90	N
4.5	45	70	1.61 ± 0.12	49.04	

A study by (Gnansounou et al., 2005) used a combination of tandem rollers and counter current diffusion on sweet sorghum and 87% of the initial sugar was extracted, which is nearly 38 percent higher compared to the results of counter current diffusion during the present study. Results of the present study were similar to a study by Noah and Linden (1989), study where a temperature of 70˚ C and higher liquid to solid ratio yielded higher sugar from sweet sorghum using a continuous counter current diffuser.

4.5 Comparison of Different Diffusion Strategies

Results indicate that the fed batch diffusion process yielded higher sugar extraction rates compared to the counter current and batch diffusion processes used in this study. This may be due to more fresh water treatments during fed batch diffusion, which increased the concentration gradient between sorghum solids and water several times during the process, which in turn increased the mass transfer and hence more sugar was extracted.

Longer contact times, higher liquid to solid ratio and higher temperatures were effective in diffusing higher sugar from sweet sorghum in all the diffusion strategies during the present study.

Figure 4.11 shows the maximum theoretical yield sugar obtained during different diffusion strategies with varying contact times at liquid to solid ratio of 4.5 and a temperature of 70°C. From the figure, it can be seen that the fed batch diffusion was more efficient compared to batch and counter current diffusion process at all contact times. Statistical analysis showed that the fed batch diffusion significantly ($p < 0.05$) yielded higher sugar compared to the other two diffusion processes with contact times of 15 and 45 minutes. Sugar yields of batch diffusion were not significantly higher compared to counter current diffusion process ($p > 0.05$) with all contact times.

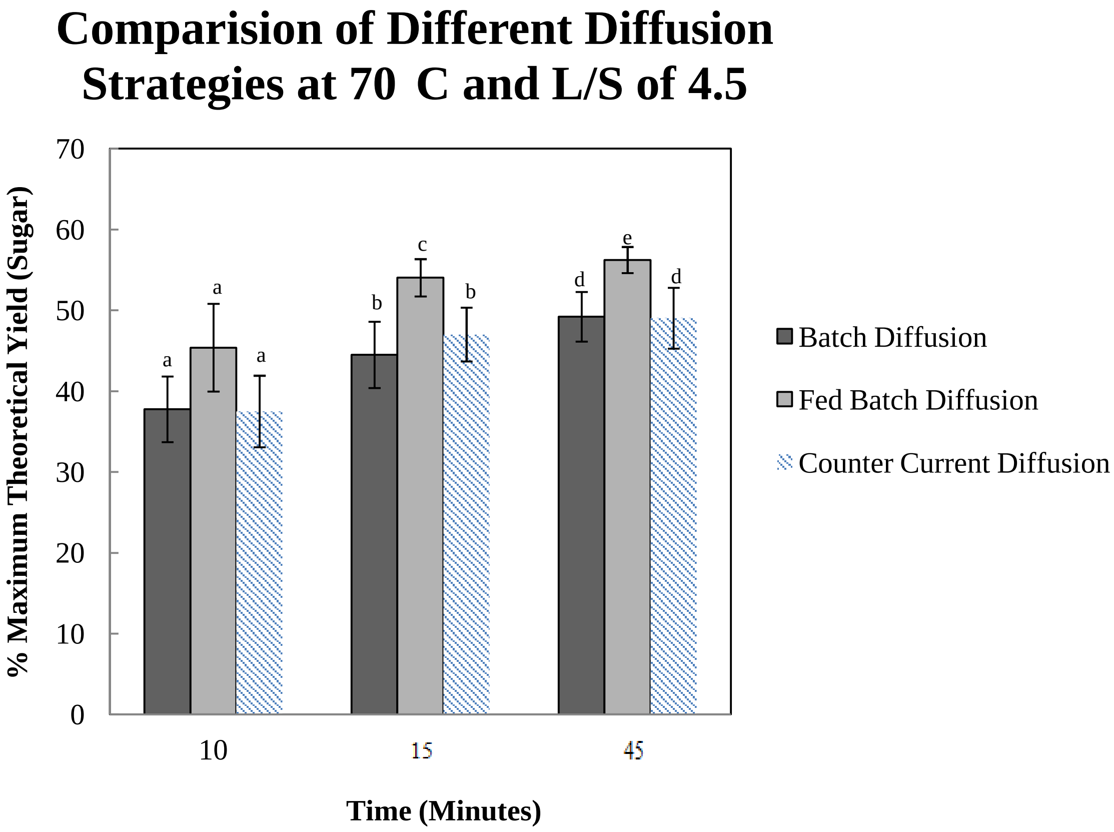


Fig. 4.11 Amount of free sugar diffused expressed as percentage of maximum theoretical yield Vs different contact times at 70°C and liquid to solid ratio of 4.5 during different diffusion strategies (n=6, error bars indicate standard error). Values at a given contact time with different letters are significantly different. Significant difference is determined at α = 0.05.

Sugar yields obtained in this study were comparable to a study by Tew et al. (2008) in which a hydraulic press was used to extract sugar from sweet sorghum. Sugar yields reported by Eshtiaghi and Yoswathana (2012) using a roller press on sugar cane were about 10% lower than the yields reported here.

Nearly 18% higher sugar was diffused using the techniques from the present study compared to the results reported by Cundiff (1992) in which a screw press was used to extract sugar from whole stalks of sweet sorghum.

CHAPTER V

CONCLUSIONS

BATCH DIFFUSION

- An increasing trend in the sugar yield was observed when L/S was increased from 1.5 to 3 and 4.5. Statistical analysis showed that this effect on batch diffusion was insignificant ($p > 0.05$).
- Contact time between sorghum solids and liquid affected the extraction efficiency. Higher sugar was achieved with longer contact times. However, this trend was significant only at L/S of 4.5 and contact time of 45 minutes compared to 10 minute contact time. The same pattern was observed at both temperatures of 60°C and 70°C.
- There was a trend of higher sugar extraction at 70°C compared to 60°C, but the difference was not statistically significant ($p > 0.05$).
- Shaking had a significant effect on the batch diffusion process. Results showed nearly 10% higher sugar compared to the non-shaking environment.

FED BATCH DIFFUSION

- Significantly higher sugar yields were observed at higher liquid to solid ratios during fed batch diffusion of sweet sorghum.
- Contact time between sorghum solids and liquid significantly affected the fed batch diffusion process. Significantly ($p < 0.05$) higher sugar was obtained at 45 minutes

- compared to the 10 minute and 15 minute contact times at 70˚ C and liquid to solid ratio of 1.5, and also at a liquid to solid ratio of 3 and temperatures of 60˚ C and 70˚ C.
- A temperature of 70^{o}C yielded more sugar than 60^{o}C during fed batch diffusion. Significantly higher sugar was diffused only at L/S of 3 and contact times of 15 and 45 minutes ($p < 0.05$).
- A mixing environment significantly affected fed batch diffusion in extracting more sugar compared to a non-mixing environment. Significantly higher sugar was diffused with shaking in all conditions except for L/S of 1.5 and a temperature of 60° C.

COUNTER CURRENT DIFFUSION

- Counter Current diffusion of sweet sorghum yielded the highest amount of sugar at L/S of 4.5 compared to 3 and 1.5. Statistical analysis revealed that L/S significantly ($P < 0.05$) affected sugar diffusion with a 15 minute contact time at 70°C and at 60°C it was significant at the 45 minute contact time.
- 70° C yielded higher sugar compared to 60° C at all contact times and liquid to solid ratios. This effect was significant only at L/S of 3 and contact times of 15 and 45 minutes.
- Among the three time parameters that were tested, a diffusion time of 45 minutes yielded the highest amount of sugar compared to the10 and 15 minute contact times at most of the L/S ratios and temperatures that were tested. Significantly ($P < 0.05$) higher sugar was diffused with the 45 minute contact time compared to the 10 minute contact time at L/S of 3 and 4.5 at 70° C.

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