



Non-catalytic steam hydrolysis of fats and oils
by Richard Charles Archuleta

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in
Chemical Engineering
Montana State University
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Abstract:

The hydrolysis of fats or oils with water produces fatty acids and glycerol. Currently, the countercurrent, continuous, high pressure, high temperature, liquid phase Colgate-Emery process, which uses a catalyst to promote the reaction, is the state-of-the-art method of fat hydrolysis. Non-catalytic steam hydrolysis is a possible alternative. Significant degrees of hydrolysis may be achieved by continuously sparging superheated steam through high temperature fat at atmospheric pressure.

A "bench-scale" hydrolyzer was designed and constructed to investigate the feasibility of steam hydrolysis and was tested with soybean oil and beef tallow. Each of the five stages in the 316 Stainless Steel, 1.77 inch inside diameter, 18-3/4 feet high hydrolyzer included a riser, a downcomer, and a steam dispersion plate similar to a distillation column. Investigations of the degree of hydrolysis were conducted at various temperatures and fat/steam feed ratios. The compositions of the overhead and bottoms products as well as stage liquid samples were analyzed.

Soybean oil was not a good feed source because its highly unsaturated fatty acids polymerized at high temperatures. The degrees of hydrolysis achieved using tallow were 15% at 280°C and 35% at 300°C at a tallow-to-steam feed ratio of 4.2. At a feed ratio of 9.2, the degree of hydrolysis was 21% at 300°C. Decomposition of glycerol was strongly evident at 325°C but not at lower temperatures. Over 95% fatty acids were present in the readily separated organic portion of the overhead product.

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This thesis has been read by each member of the thesis committee and has been found to be satisfactory regarding content, English usage, format, citations, bibliographic style, and consistency, and is ready for submission to the College of Graduate Studies.

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ABSTRACT

The hydrolysis of fats or oils with water produces fatty acids and glycerol. Currently, the countercurrent, continuous, high pressure, high temperature, liquid phase Colgate-Emery process, which uses a catalyst to promote the reaction, is the state-of-the-art method of fat hydrolysis. Non-catalytic steam hydrolysis is a possible alternative. Significant degrees of hydrolysis may be achieved by continuously sparging superheated steam through high temperature fat at atmospheric pressure.

A "bench-scale" hydrolyzer was designed and constructed to investigate the feasibility of steam hydrolysis and was tested with soybean oil and beef tallow. Each of the five stages in the 316 Stainless Steel, 1.77 inch inside diameter, 18-3/4 feet high hydrolyzer included a riser, a downcomer, and a steam dispersion plate similar to a distillation column. Investigations of the degree of hydrolysis were conducted at various temperatures and fat/steam feed ratios. The compositions of the overhead and bottoms products as well as stage liquid samples were analyzed.

Soybean oil was not a good feed source because its highly unsaturated fatty acids polymerized at high temperatures. The degrees of hydrolysis achieved using tallow were 15% at 280°C and 35% at 300°C at a tallow-to-steam feed ratio of 4.2. At a feed ratio of 9.2, the degree of hydrolysis was 21% at 300°C. Decomposition of glycerol was strongly evident at 325°C but not at lower temperatures. Over 95% fatty acids were present in the readily separated organic portion of the overhead product.

CHAPTER 1**INTRODUCTION**Industrial Uses for Fat and Oil Hydrolysis Products

The hydrolysis of fats and oils produces several important industrial chemicals. These products include fatty acids, monoglycerides, diglycerides, and glycerols. Depending on the type of fat or oil feed and the conditions of the reaction itself, a wide array of product possibilities can be achieved.

According to 1990 statistics, the United States alone consumed over a billion pounds of fatty acids [1]. The primary sources for fatty acids were animal fats, coconut oil, and vegetable oils [2]. It has been also estimated that over 185 million pounds of mono- and di- glycerides were produced [3]. Finally, approximately 310 million pounds of glycerol were produced in 1990 by hydrolysis of fats and oils [4].

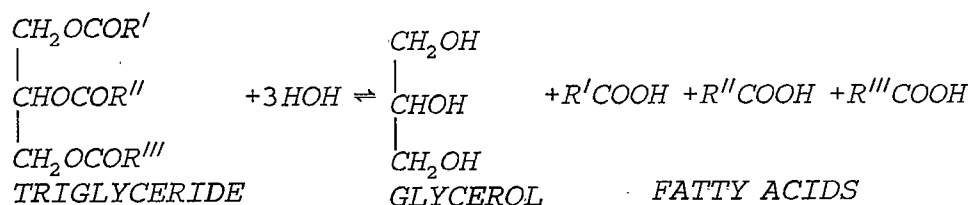
Fatty acids are manufactured for several uses. Products in which purified fatty acids are used in large quantities include soaps, synthetic surface-active agents, plasticizers, polymers, lubricating greases, and many rubber applications. The largest consumption of fatty acids is in the areas of soaps and surfactants [5].

Monoglycerides and diglycerides are specialty chemicals because performance rather than price is the main factor for purchasing decisions. Applications are limited mostly to the food industry. However, monoglycerides and diglycerides are also used in cosmetics, surfactants, and protective coatings [6].

Glycerol products are encountered in many areas. Glycerols are used in solvents, plasticizers, emollients, sweeteners, cosmetics, liquid soaps, liqueurs, copying inks, and antifreeze for automobiles to name a few [7]. Glycerol produced from hydrolysis is not the only source of glycerol for the market, but it contributes significantly.

The Chemistry of Fat and Oil Hydrolysis

The hydrolysis of fat and oil triglycerides to free fatty acids and glycerol requires conditions which promote triglyceride and water miscibility. The reaction is not simple because it proceeds in stages and is reversible. The general chemical reaction formula for fat and oil hydrolysis is:

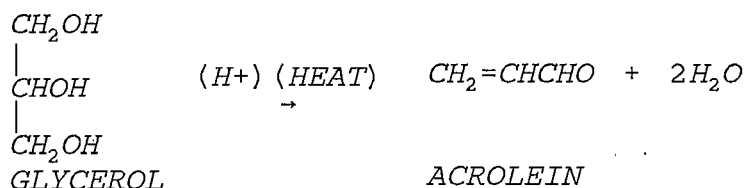


where R', R'', and R''' are straight chain compounds containing from three to eighteen carbons. Only acids

containing an even number of carbons are present in substantial amounts. Acids may be saturated or partially unsaturated depending on the fat. The proportions of the various acids vary from fat to fat; each fat has its characteristic composition, which does not differ very much from sample to sample.

Hydrolysis products must be removed from the area of reaction to ensure a forward rate of reaction. A high degree of hydrolysis is ensured in the standard industrial process by using a large excess of water and withdrawing the aqueous glycerol-rich phase and replacing it with fresh water. High temperatures also accelerate hydrolysis [8]. If hydrolysis does not completely remove the fatty acids from a triglyceride molecule, the resulting product is either a monoglyceride (one remaining fatty acid esterified on glycerol) or a diglyceride (two remaining fatty acids esterified on glycerol).

At high temperatures, there is a side reaction that results in the decomposition of glycerol into acrolein. The most sensitive indication of acrolein is its lachrymatory odor. The dehydration of glycerol to yield acrolein involves acid-catalyzed dehydration and keto-enol tautomerization [9]. The chemical reaction for decomposition is:



The acid catalyst is provided by fatty acids from the reaction.

CHAPTER 2**CURRENT INDUSTRIAL HYDROLYSIS TECHNOLOGY**The Colgate-Emery Process

Current industrial fat hydrolysis utilizes primarily the Colgate-Emery process which was developed in the 1940's. There has been little change in the technology and the process is widely used today. This continuous high temperature fat splitting process employs a countercurrent reaction in a pressure tower with internal heat exchange resulting in efficiencies of 98%. Using high temperatures and pressures permits relatively short reaction times. Countercurrent fat and water flow produces high degrees of hydrolysis without the use of a catalyst, but plant capacity can be increased by adding a small amount of catalyst to the reaction. However, catalysts promote decomposition of glycerol into acrolein which is undesirable [10].

The well insulated reactor tower is approximately 20-48 inches in diameter, 60-80 feet high, made of solid 316 stainless steel to withstand operating pressures of over 700 psi. Fat is introduced by means of a sparge ring about 3 feet from the bottom, with a high pressure feed pump. Water is introduced near the top of the column at a ratio of 40-50% of the weight of the fat which is about 21 moles of water feed

per mole of fat feed. As the ascending fat nears the reaction temperature of approximately 260°C, hydrolysis proceeds rapidly. The approximate residence time of both the fat and water phases through the process is 2 to 3 hours. Temperature independence of the point of equilibrium is an indication of a zero heat of reaction in the Colgate-Emery process [11].

Reaction Kinetics of Hydrolyzing Fats and Oils

There is a generally accepted mechanism for fat hydrolysis. The hydrolysis of fats and oils to fatty acids is sequential from the tri- to the di- to the monoglyceride and finally to the glycerol. It has been observed that in the initial stages of the reaction, an oil/water emulsion forms and the reaction proceeds slowly. That is, initially, the reaction takes place at the interface of the organic and aqueous phases. As the reaction continues, the emulsion breaks down and the reaction rate increases significantly. At this point, the fatty acid content in the oil increases causing the solubility of water in the organic phase to increase. The reaction becomes more homogenous in the organic phase. Finally, as the degree of hydrolysis increases further, the net reaction rate begins to decrease because the reesterification reactions begin to occur at significant rates. In the Colgate-Emery process, the hydrolysis forward reaction is driven nearly to completion by preferential

extraction of the glycerol from the organic phase into the water phase [12].

The solubility of water in fats and oils is almost non-existent at room temperature and very low at higher temperatures. However, it is difficult to determine the solubility of water in fats at high temperatures because of the effects of hydrolysis. The solubility of water in fatty acids is much higher than in mono-, di-, or triglycerides. This helps to explain the stages of the hydrolysis mechanism at higher degrees of reaction.

CHAPTER 3

THE FEASIBILITY OF STEAM HYDROLYSIS

Steam Hydrolysis of Fats and Oils

The Colgate-Emery process is limited since it requires a reaction time of 2-3 hours. There is a significant cost of energy required to recover glycerol from the reaction products. To decrease the reaction time and the amount of energy required to separate and purify the products, Kenneth Lunde has investigated the possibility of steam hydrolysis in contrast to water hydrolysis [13].

The steam hydrolysis of fats and oils involves the contact of superheated steam at a pressure of approximately one atmosphere with a fat or oil feed. Glycerol is removed almost entirely from the reaction mixture by vaporization rather than by extraction from the liquid organic phase to liquid water. There is not a need for a catalyst to increase the rate of reaction.

For an industrial steam hydrolysis process the fat or oil feed would initially be deaerated and then heated to reaction temperature. The feed would then be pumped into the top of the reaction column. Steam at a temperature equivalent to the entering fat feed supply would be sparged into the base of the column. The reactor operates essentially adiabatically since

there is insignificant heat of reaction. The mechanism of the reaction and the solubility of water in the organic phase is similar to high pressure, high temperature water hydrolysis. As steam ascends through the hydrolyzer and liquid fat or oil descends, the reaction proceeds, and glycerol is stripped away from the reaction by the ascending vapor, driving the reaction forward. Almost complete hydrolysis should occur.

Steam ascending through the column would strip glycerol from the system resulting in a shorter glycerol residence time for steam hydrolysis over the Colgate-Emery process. Because the density of steam is much less than the density of liquid water, a faster volumetric flow rate of vapor can be provided in steam hydrolysis over the Colgate-Emery process assuming that the same relative mass feed rates of fat and water are utilized.

A high reaction temperature may be necessary for a non-catalyzed steam hydrolysis reaction, but less decomposition of products should occur in the reaction vessel due to the shorter residence time of the glycerol in the reactor and the absence of a catalyst which promotes the glycerol decomposition reaction. Because the product streams should show little decomposition, less processing and energy may be necessary to purify the products.

Previous Experimental Results on Steam Hydrolysis

Preliminary work has been performed on the feasibility of steam hydrolysis. Kenneth Lunde has investigated the possibility of catalyzed steam hydrolysis [13]. Some important conclusions were reported that affect the design of the experimental steam hydrolyzer.

Some reactions are limited by chemical kinetics at low temperatures, but with elevated temperatures the rate constant is sufficiently large that the reaction rate is limited by mass transfer. The "Carad" experiments by Lunde were performed to ascertain whether this is the case in the hydrolysis of fats and oils [13]. An investigation was made to increase the solubility of water into the organic phase by use of superheated steam at relatively low pressures in the presence of a catalyst. Tallow at 200-280°C was 16% hydrolyzed at only 10-30 second residence time through a specially designed parallel flow pipe reactor using a zinc oxide catalyst. A second pass of the oil phase through the system resulted in 70% hydrolysis. The experiments indicated that even at high temperatures, the reaction rate is limited by chemical kinetics. It was also concluded that catalysts accelerate the decomposition of glycerol, tallow, and the fatty acids of tallow. This decomposition limits the operating temperature and, therefore, the reaction rate [13].

Research Objectives

1. Investigate feed rate and temperature operating parameters for a bench-scale hydrolyzer.
2. Analyze stage and overall product samples for the extent of hydrolysis and determine if decomposition of the products is apparent.
3. Gain initial insight into the commercial feasibility for the non-catalyzed steam hydrolysis of fats.

CHAPTER 4**THE BENCH SCALE HYDROLYZER**Background

To investigate the feasibility of non-catalytic steam hydrolysis, a bench scale reactor has been designed and constructed. The five tray hydrolyzer reactor is approximately 18 feet long and approximately 1-1/2 inch diameter. A steam source is provided from a boiler, and a tallow feed supply is used.

Investigation of various fat to steam feed ratios is desired to understand the feasibility of the process. Further, there is a need to analyze the content of overhead and bottoms products to understand the extent of the reaction. Finally, by adjusting the temperature of the reaction, the extent of the reaction can be determined for various temperature ranges. The presence of decomposition can also be determined by sensing for an acrolein odor.

The bench-scale hydrolyzer allows several variables to be tested. A range of temperatures, fat feed rates, steam feed rates, and fat/steam feed rate ratios can be tested and a range of outputs observed. These observations can be used to establish the feasibility of an industrial sized process. Investigations of various grades of stainless steel to

determine an appropriate hydrolyzer construction material is important. Fatty acids are corrosive which can lead to poor initial color of fatty acids or poor stability during further processing [14].

The bench scale hydrolyzer was constructed in Ryon Lab 2 at Montana State University. The hydrolyzer was constructed during the Spring and Summer of 1990 with minor design modifications made later. At the current time, investigation of its operation continues.

There are five main components which comprise the design of the hydrolyzer. These are a steam source, a fat or oil feed source, the reactor itself, a bottoms collection system, and a system to condense and collect overhead vapor products as liquids. A heating and temperature control system is necessary for the entire process. The discussion which follows describes the components of equipment which are included in the overall system diagramed in Figure 1.

The Steam Feed System

The steam feed system is illustrated in Figure 2. The system includes a steam boiler, a calibrated fine metering needle valve, a steam superheater, and a nitrogen purge supply. This section provides a calibrated superheated steam feed or nitrogen purge supply to the hydrolyzer.

The boiler is a 316 Stainless Steel (SS) tank with 3/16 inch thick walls which is 45 inches high with a diameter of 8

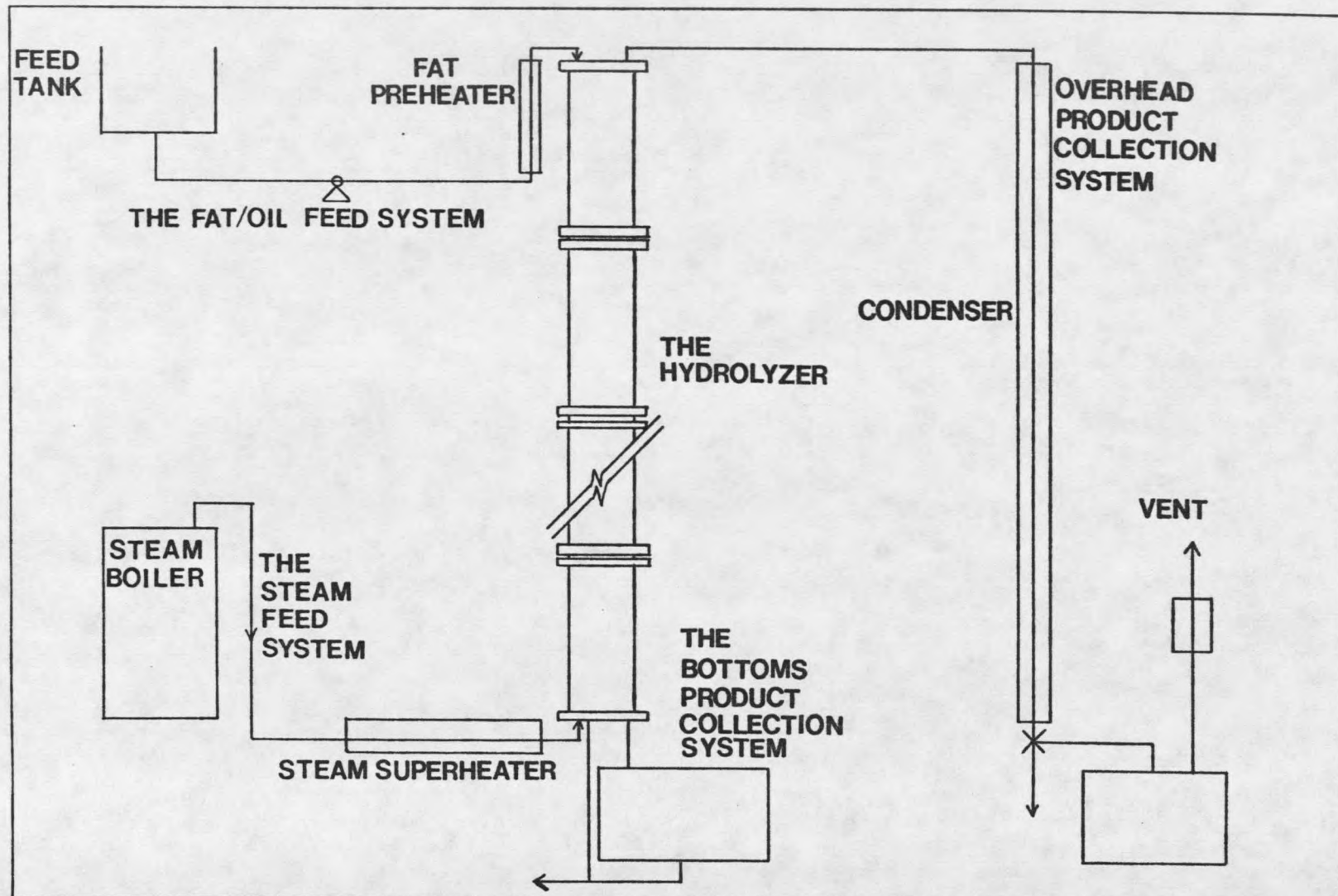


FIGURE 1. THE BENCH SCALE HYDROLYZER

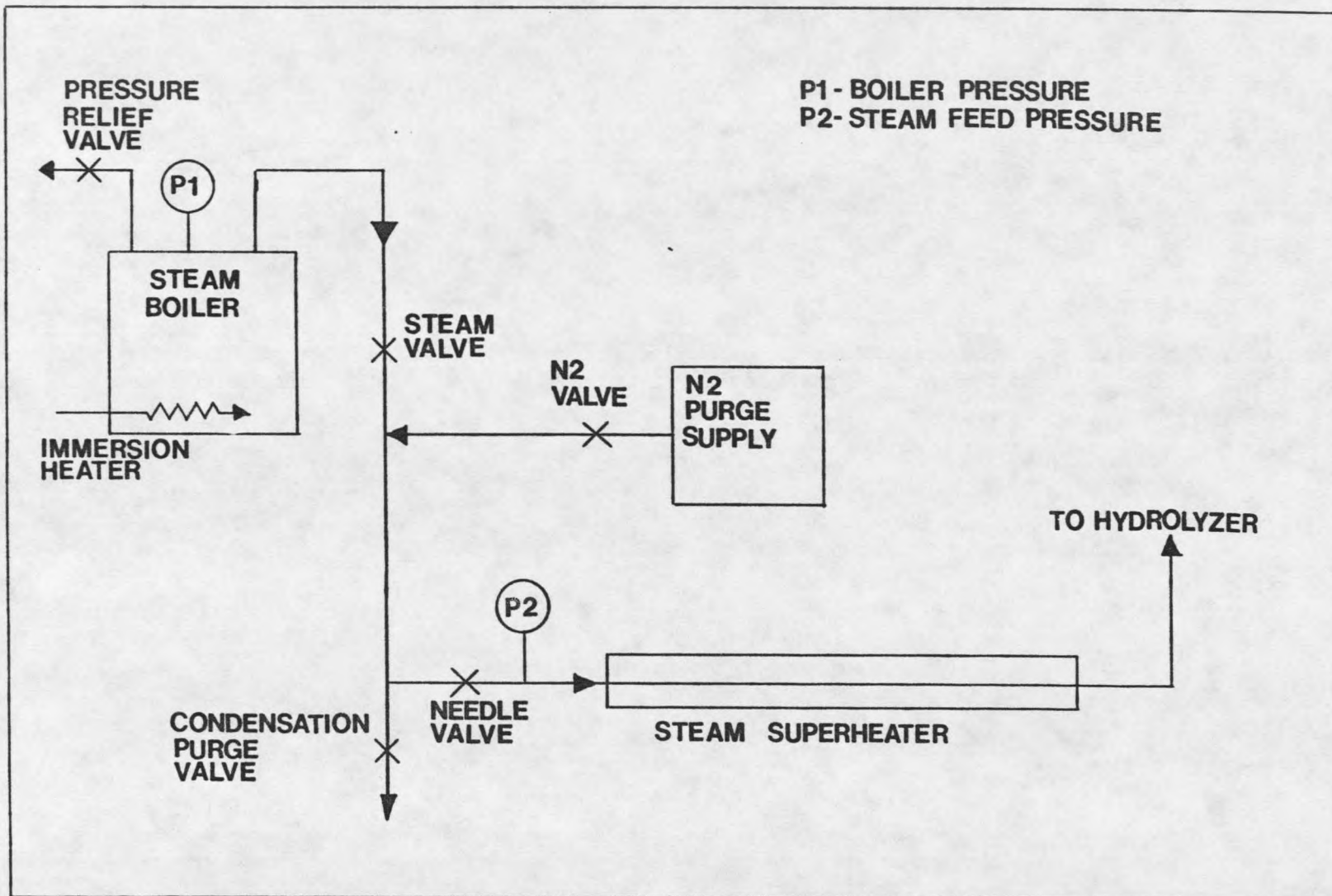


FIGURE 2. THE STEAM FEED SYSTEM

inches. By partially filling the vertical tank with distilled water and applying thermal energy to bring the temperature of the boiler to 130°C, a 30 psig steam source is made available. The boiler is equipped with a pressure relief valve which is set for safe operation of the unit.

Thermal energy is supplied using a 240 volt Chromalox Screwplug Immersion heater located 3-1/2 inches up from the bottom of the boiler. The water temperature is monitored by two thermocouples located in a thermocouple well located 8 inches up from the bottom of the boiler. Energy supply to the heater is controlled by an on-off temperature controller attached to one of the thermocouples. Steam pressure is monitored by a Bourdon gauge at the top of the boiler.

Steam occupies the top space in the boiler. Steam exits the boiler through a shut off valve. Condensation in the steam is purged to the outside atmosphere through a shut off valve in an open branch of a union tee ahead of a metering valve. The steam then flows through the fine metering valve.

A purge gas supply for the reaction system can be provided upstream of the needle valve from a cylinder of high pressure nitrogen. The nitrogen is used to purge process lines and the reactor. As will be discussed later, it was also used for determining a stable fluid flow operating range for the reactor.

The calibrated Nupro fine metering needle valve controls the flow rate of the steam to the reactor. A thermocouple

attached at the needle valve monitors its temperature. The pressure of the steam supply to the reactor is measured by a Bourdon gauge downstream of the needle valve which indicates the pressure drop across the column to the atmosphere at the exit of the overhead condenser.

Calibration of the steam flow rate was performed by adjusting the needle valve to each of a series of settings and allowing the steam to pass through the reactor to the overhead condenser. Steam condensate was collected in a graduated cylinder at the exit of the overhead condensate line. Steam condensate collections were made for at least 30 minutes. The resulting calibration for the steam feed rate is shown in Figure 3 for steam feed rates from 0.5 g/min to 4.9 g/min.

Before the steam enters the reactor, it is superheated to the reaction temperature in a 10 foot long 3/4 inch, Schedule 80 316 SS pipe. The pipe is wrapped with two 400 watt heating tapes in series. The upstream tape increases the steam temperature to near the operating temperature. The steam exit temperature downstream of this tape is manually controlled by a variac in the electric supply to this tape. The power supply to the downstream heating tape is regulated by an on-off temperature controller which establishes the final steam temperature. Thermocouples attached to the pipe at the end of each heating tape monitor the steam temperatures at those locations. A third thermocouple is attached to the end of the

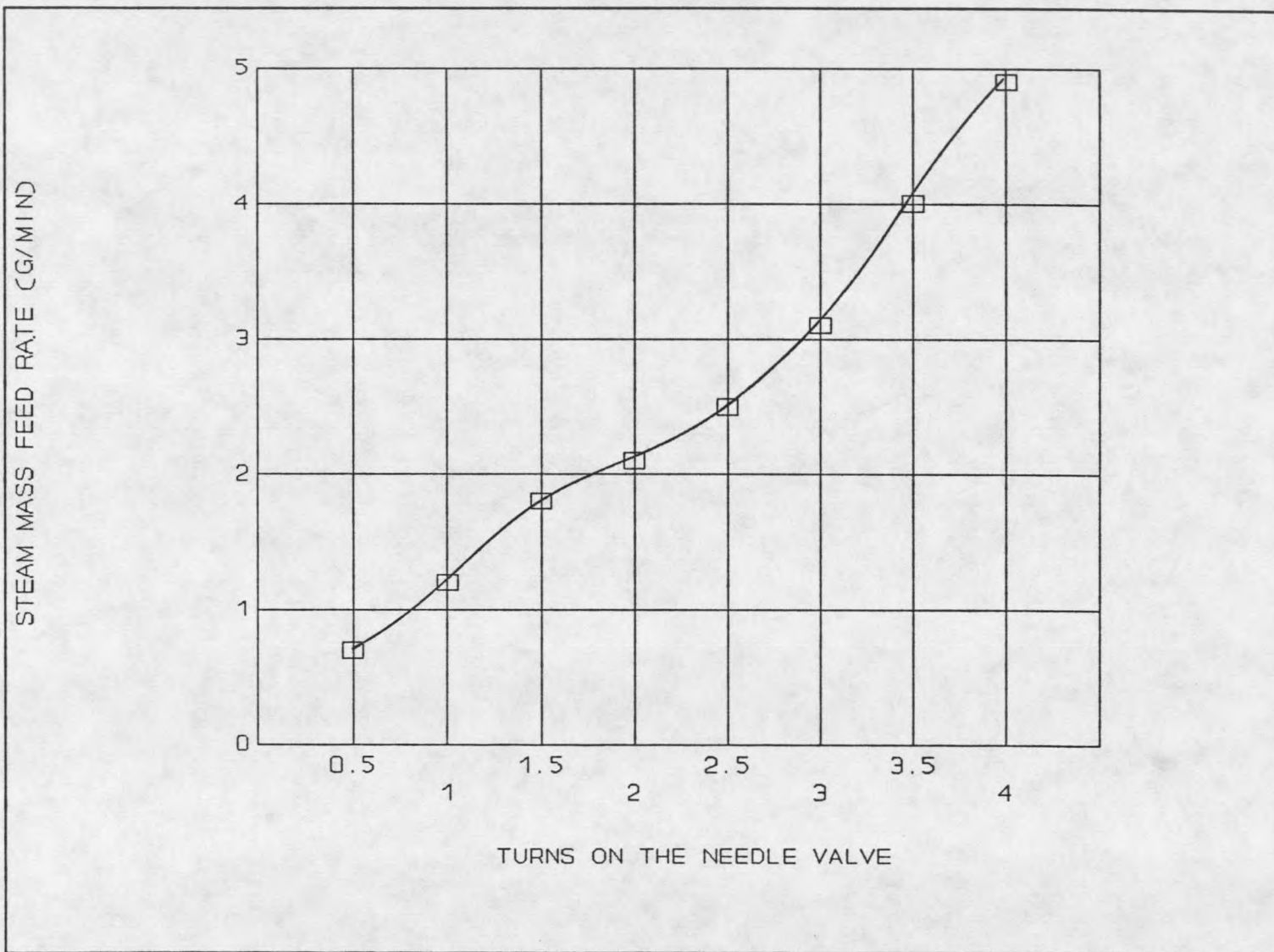


FIGURE 3. NEEDLE VALVE CALIBRATION OF STEAM FEED

downstream heating tape to provide the signal to the steam supply temperature controller.

The Fat/Oil Feed System

The fat/oil feed system is designed to supply any of several different preheated fat and oil materials to the hydrolyzer. As illustrated in Figure 4, the fat/oil feed system consists of a heated feed tank, a calibrated metering pump, and a fat preheater.

Since beef tallow, the typical feed material, has a softening point higher than room temperature, it is necessary to melt the feed. The tallow is melted in a 15 inch long, 12.5 inch diameter 316 SS feed tank that is open at the top. The outside of the tank is wrapped with heating tape and is temperature controlled. Two thermocouples are attached to the side of the tank near the bottom. One monitors the tank temperature, and the other provides a signal for the temperature controller. The fat feed tank is approximately three feet off the ground to provide a head for the feed pump. Tallow exits the tank through a 1/4 inch 316 SS tube connected to the center of the bottom of the tank. The tube intake is raised one inch above the bottom of the tank to avoid allowing water that may be present in the feed to flow into the system. The tube to the feed pump is wrapped with heat tracing cable to prevent fat from hardening in the feed line.

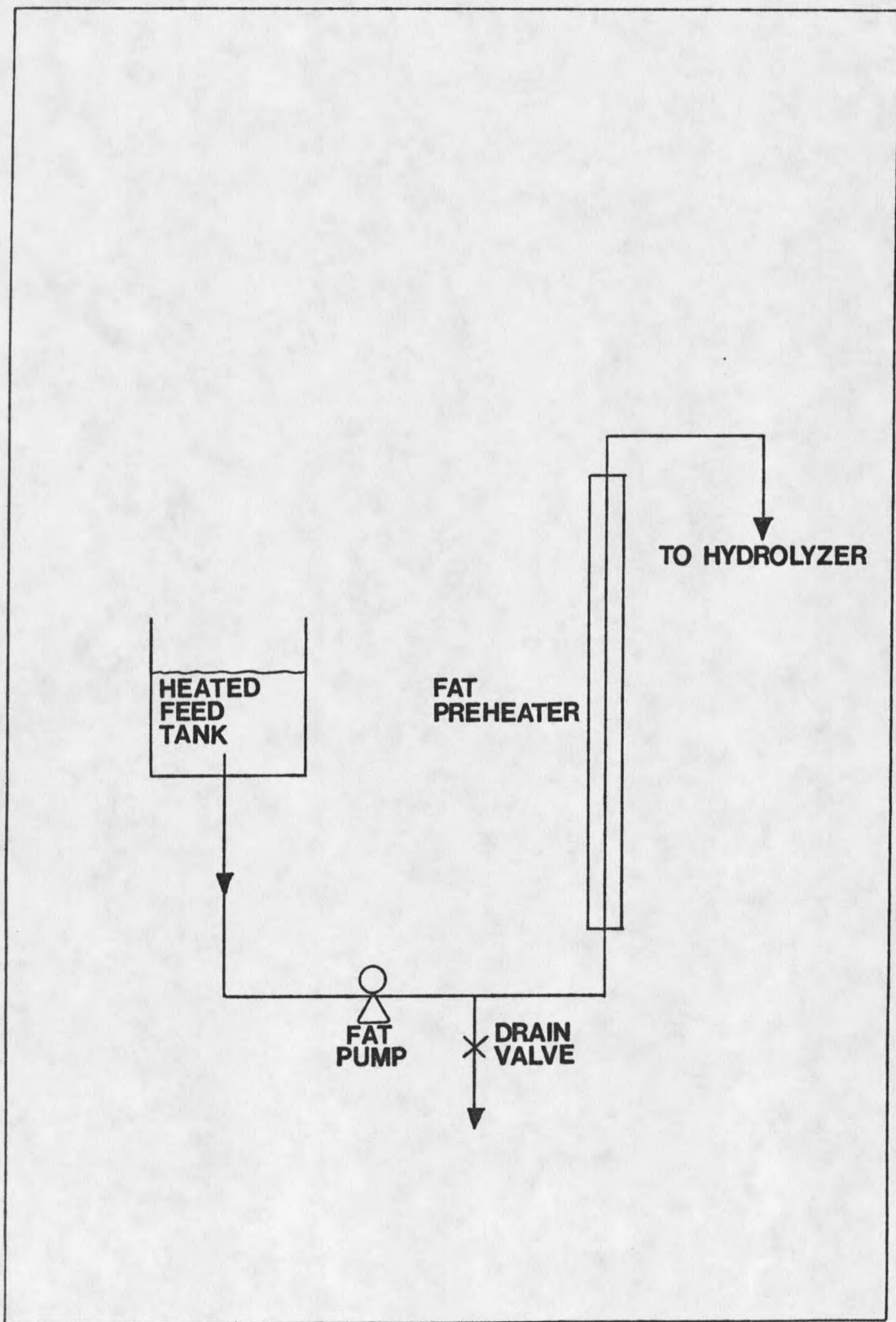


FIGURE 4. THE FAT/OIL FEED SYSTEM

A Simplex Milroyal Type B controlled volume, positive displacement reciprocating pump with a 5/16 inch plunger was used to feed the tallow from the supply tank to the top of the reactor. The pump was calibrated for a range of fat feed rates by turning a control knob which adjusted the plunger stroke and by collecting the feed for at least five minutes in a 400-ml preweighed beaker. By weighing the beaker contents, the mass flow rate of the feed could be determined for each pump setting. The results of the tallow feed calibration are reported in Figure 5 for fat feed rates from 5 grams/min to 90 grams/min.

Heat tracing cable is wrapped around the plunger area of the pump to prevent tallow from hardening within the pump. The tallow is pumped through 1/4 inch 316 SS tubing of approximately 10 feet in length to the fat preheater. A drain valve is used for purging the feed line when the process is shut down. A thermocouple is located midway along this tubing to monitor its temperature to ensure the tallow is above its hardening temperature.

The 1/4 inch tube is connected to a thick walled 3/4 inch Schedule XX, 316 SS pipe which extends the remainder of the distance to the top of the reactor. There are two 400 watt heating tapes wrapped on this pipe. The upstream tape extends half way along the pipe and the second heat tape along the downstream portion of the pipe. The first tape is used to warm the tallow to a temperature near the reactor operating

