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HYDROGENATION OF OILS

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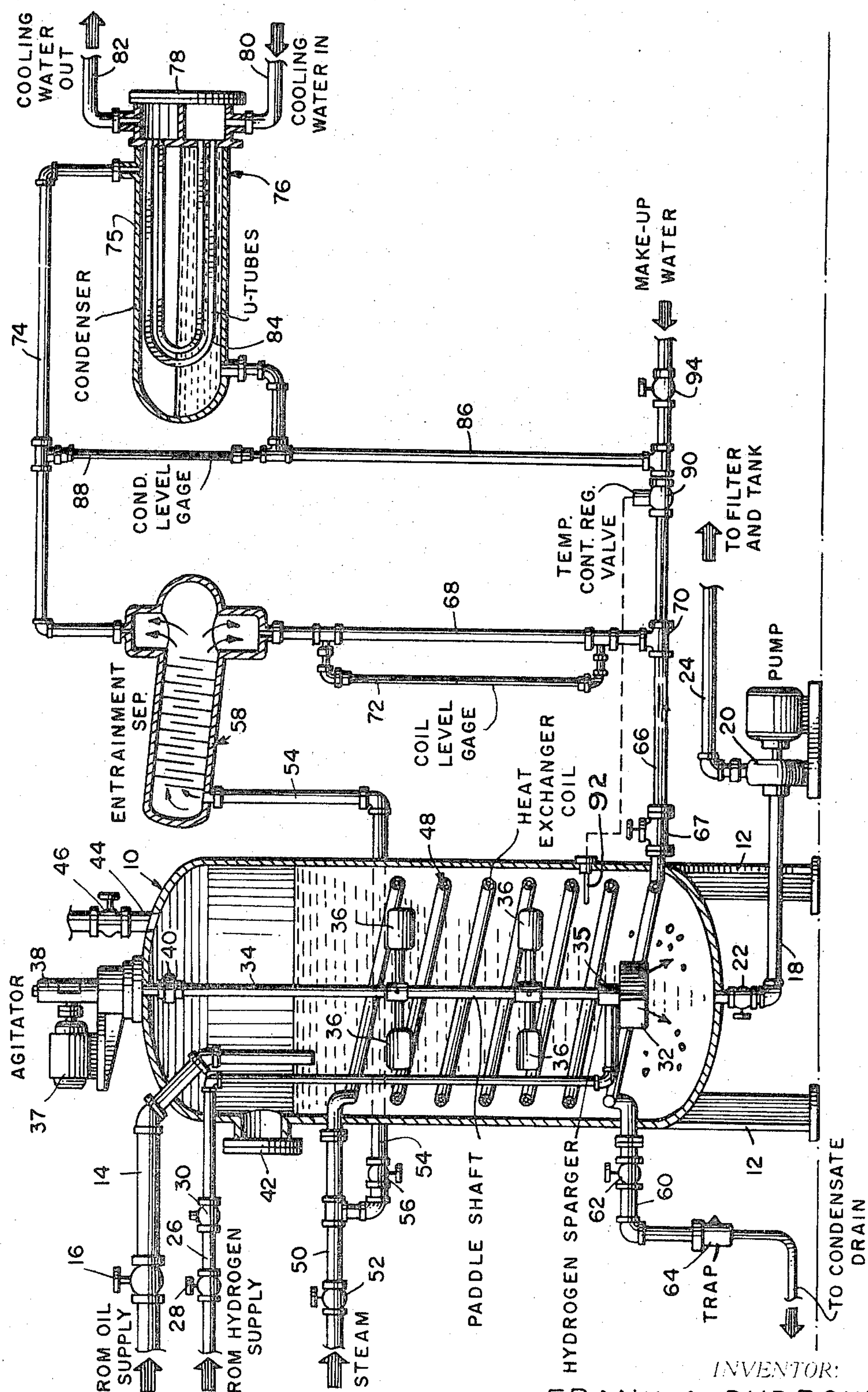

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HYDROGENATION OF OILS

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ABSTRACT OF THE DISCLOSURE

A method and apparatus are provided for control of the temperature of a fluid reaction mass. Heat exchanger means in the reaction vessel contains a body of coolant which is vaporized to absorb heat. A closed coolant condensation system communicates with the heat exchanger means to remove heat as it is generated.

THE INVENTION

The present invention relates to a new and improved method and apparatus for providing controlled temperature in a reaction mass and is especially useful in hydrogenation of unsaturated oils. Demand for margarines and shortenings high in unsaturated fats has placed great emphasis on selective hydrogenation processes for producing the same. The exact conditions necessary to achieve a desired final product from a given type of raw material in a particular system are normally determined by trial and error, and once the conditions and parameters have been established it is very important to provide for a high degree of control repeatability in order to insure a continuous high quality and uniformity of the finished product. For example, when vegetable oil such as soybean oil from a particular source is hydrogenated to produce margarine, the temperature of the oil during the process must be maintained within a narrow desired range to achieve uniformity of quality and the desired degree of selective hydrogenation.

In prior art systems, temperature control of the reaction was erratic and difficult. According to conventional practice, a heat exchanger coil was designed for initially heating the oil with steam to a temperature sufficiently high to enable the reaction to progress at a practical rate; thereafter the coil was used for cooling the oil as the reaction became exothermic. The economics of equipment utilization required the reaction to be run as rapidly as possible, but this aggravated the cooling problems during the highly exothermic initial hydrogenation phase. The reaction was usually performed at an oil temperature from about 240° F. to about 450° F. The cooling water however flashed into steam at around 212° F. which temperature, being far below the usual hydrogenation temperature made accurate control most difficult. Moreover, when cooling at a high rate was required, operators became apprehensive and admitted too much water and the excess caused undesired deviations in oil temperature. In addition as the water was violently flashed into steam, severe vibration and hammering occurred in the coil, resulting in physical damage and frequent coil failures. Wide variations in temperature commonly encountered caused high rates of erosion and scaling up of the internal surfaces of the coil thus further adding to the difficulty of accurately maintaining and controlling the temperature of the hydrogenation process.

The present invention provides a new and improved method and apparatus for the selective hydrogenation of unsaturated oils, wherein the temperature of the process is accurately controlled and maintained at a predetermined level with very little variation. The invention affords a high degree of controlled repeatability of the hydrogenation process for a given raw material and final product, so that repeated batches can be processed with uniformity

of selective hydrogenation. The invention also provides a degree of temperature control previously unattainable.

The apparatus of the present invention includes a heat exchanger coil in the reaction vessel and a closed, coolant condensing system is in communication with the heat exchanger. The condensing system can be maintained at any desired operating pressure, either subatmospheric or superatmospheric, for example from about 1 p.s.i.a. to about 450 p.s.i.a. Maintenance of superatmospheric pressure on the cooling system enables the coolant to be near the desired operating temperature without vaporizing so that there may be a large quantity of coolant in the coil. Reduction in condensing system pressure causes immediate vaporization so that the time for a temperature change is reduced and much closer temperature control of the hydrogenation process can be established. At subatmospheric pressure, the vaporization temperature is sufficiently low that the reaction mass may be chilled.

It is an object of the invention to provide a new and improved method and apparatus for controlling the temperature of a reaction mass, particularly during an exothermic reaction.

Another object is the provision of a new and improved method and apparatus for controlling the temperature of a hydrogenation process.

Yet another object of the invention is the provision of a new and improved method and apparatus for producing food products having controlled polyunsaturates therein by the hydrogenation of unsaturated oils.

Another object of the present invention is the provision of a new and improved method and apparatus for the controlled hydrogenation of unsaturated oil wherein the vaporization of coolant from the heat exchanger is controlled by a change of pressure in the coolant system.

Another object of the present invention is the provision of a new and improved method and apparatus for the controlled hydrogenation of unsaturated oils wherein the reaction temperature of the oil is regulated and controlled by the amount of condensing surface in a condenser which is exposed above the surface level of the condensate in the condenser.

Yet another object of the present invention is the provision of a new and improved apparatus for the controlled hydrogenation of unsaturated oils having a closed, coolant recirculation system wherein a condenser is provided and coolant liquid condensate is metered from the condenser into the heat exchanger through a temperature controlled regulator valve sensitive to the temperature of the oil in the reaction vessel.

The foregoing and other objects and advantages of the present invention are accomplished in a new and improved apparatus for the controlled hydrogenation of unsaturated oils which comprises a reaction vessel for holding a batch of oil and means for introducing hydrogen into the oil below the surface thereof. Heat exchanger means is provided in the vessel for initially heating the oil and thereafter extracting heat from the oil as the exothermic heat of reaction takes place. A closed, coolant condensation system is provided in communication with heat exchanger means to provide for removal of heat at a controlled rate from vaporized coolant as the exothermic reaction proceeds in order to maintain the temperature of the oil at a substantially predetermined value. Cooling takes place in response to a reduction in pressure in the closed condensation system. In order to produce a specific quality or type of margarine having a certain selective hydrogenation, it is generally desirable to hold reaction temperature within a range of plus or minus 7° F. and preferably plus or minus 3° F. of the optimum temperature. A fixed temperature, for example, 370° F., may be selected or a time-temperature schedule may be followed.

If successive batches of soybean oil from the same source are processed at the same temperature, pressure and hydrogen flow rate, a consistent quality final product will generally result. The present invention provides a high degree of controlled repeatability for such hydrogenation processes.

For better understanding of the present invention, reference should be had to the following detailed description when taken in conjunction with the claims, in which:

FIG. 1 is a schematic diagram illustrating a new and improved apparatus constructed in accordance with the features of the present invention for the controlled hydrogenation of unsaturated oils.

Referring to the drawings, a pressure vessel **10** of the desired capacity is supported in an upright position on a plurality of legs **12** from the floor or other support. Unsaturated oil to be hydrogenated in the process is supplied to the vessel through an inlet supply line **14** having a control valve **16** therein. Catalyst is generally premixed with a portion of the oil before delivery to the reaction vessel but it may be added separately through a port **42**. On completion of a batch, the hydrogenated oil is pumped out of the vessel **10** via an outlet line **18** and motor driven pump **20**. A suitable control valve **22** is provided in the vessel outlet line **18** and a pump discharge line **24** is provided to pass the oil to a filter system (not shown) for removing the catalyst from the product.

Hydrogen gas for the hydrogenation process is supplied from a source (not shown) through a supply line **26** having a control valve **28** and a pressure regulator valve **30** therein. The hydrogen is introduced into the batch of oil in the vessel **10** well below the surface thereof through a sparger apparatus **32** which is mounted at the lower end of an agitator shaft **34**. The rotating shaft carries a plurality of paddles **36** for agitating and circulating the oil. The hydrogen supply line **26** is connected to a sparger sleeve **35** around the shaft, and the hydrogen gas is directed downwardly from the sleeve and outwardly by the sparger into the oil. The agitator is driven by an electric motor **37** and gear reducer **38** mounted on top of the tank and coupled to the agitator shaft by a detachable coupling **40**.

The reaction vessel **10** includes a vent line **44** with a valve **46** therein for removal of unwanted gases from the head space in the vessel above the level of the oil.

In accordance with the present invention, a heat exchanger coil **48** is mounted in the lower portion of the vessel **10** generally below the normal upper surface level of a batch volume of oil to be treated. The upper end of the coil **48** is connected to a steam supply line **50** having a control valve **52** therein for admitting steam into the coil during the initial phase of operation to heat the oil prior to the introduction of hydrogen.

Coil **48** is connected via a T to a vapor line **54** having a control valve **56** therein, and the vapor line is connected to one end of an entrainment separator **58** (shown schematically). The lower end of the heat exchanger coil **48** is connected to a discharge line **60** having a valve **62** and a steam trap **64** therein to discharge condensate during the heating step.

Condensate or coolant liquid is supplied to the lower end of the heat exchanger coil **48** through a liquid supply line **66**. Any liquid entrained in the vapor is removed in the entrainment separator **58** and is directed into the line **66** through a drain line **68** and a T **70**. The separator drain line **68** is provided with a sight gauge **72** for indicating the level of liquid in the heat exchanger coil **48**.

After removal of entrained liquid from the vapor in the entrainment separator **58**, the vapor passes out through a vapor line **74** into the shell **75** of a condenser **76**. The condenser **76** includes a pressurized outer shell **75** with a head chamber **78** at one end. Cooling water is introduced into the lower half of the head chamber through inlet line **80**, and leaves the condenser from the upper half of the head chamber **78** through an outlet line **82**. The cooling water passes from the lower half of the head chamber **78** through a bundle of heat exchanger tubes **84** in the shell which may be of the U-tube type as shown. The vapor entering the upper portion of the condenser shell passes over the U-tubes to condense and collect as shown. The flow of cooling water through the U-tubes is adequate to maintain a flooded condition in the condenser shell when desired. Condensate may be recirculated back to the liquid supply line **66** through a condensate drain line **86**. A condenser level sight gauge **88** is provided between the vapor line **74** and the condensate line **86** to indicate the height of condensate liquid in the condenser.

In accordance with the present invention, condensate is removed from the condenser shell **75** and the liquid is introduced into the lower end of the heat exchanger coil **48** from the liquid supply line **66**. The flow rate of condensate is controlled by a temperature controlled regulator valve **90**. The valve **90** is actuated in response to a temperature sensing device **92** in the vessel **10** and the device **92** is directly responsive to the temperature of the oil in the vessel. The valve opens and closes to drain more or less condensate from the condenser **76** in response to temperature variations as sensed by the temperature probe **92**.

For example, if the temperature of the oil in the vessel **10** begins to rise above the selected operating level, the temperature sensitive probe **92** sends a signal to the temperature controlled regulator valve **90** causing the valve to move toward a more open position, thereby increasing the flow rate of condensate from the condenser **76**. The converse is true should the temperature of the oil in the vessel start to drop below the selected level.

The regulator valve **90** thus exposes more or less coil surface of the condenser **76** by controlling the flow rate of condensate therefrom. This in turn varies the pressure in the closed system and controls the amount of coolant being evaporated in the coil **48** and passing out through the line **54** in vapor form.

The coil **48** in the hydrogenation vessel **10** serves in effect as an internal coolant reservoir from which coolant is vaporized as the valve 90 meters the condensate from the condenser.

In a typical margarine hydrogenation, a batch of soybean oil with catalyst is introduced into the vessel **10** through the inlet supply line **14**. The valves **56** and **67** are closed and the valve **52** is opened to admit steam at 150 p.s.i.g. into the upper end of the heat exchanger coil **48** from the line **50**. Condensate valve **62** is opened and the steam heats the oil to about 300° F., at which time the condensate valve **62** is closed. The steam now condenses in the coil and the condensate level in the coil is permitted to rise until the coil **48** is about 50% or 75% full. At this time valve **52** is closed, vapor valve **56** and condensate valve **67** are opened. Hydrogen is introduced at 5 p.s.i.g. through the sparger system **32** by opening of the hydrogen valve **28** and the agitator shaft is rotated to circulate the oil. The hydrogenation reaction raises the oil temperature to 400° F. in about one hour. As the oil temperature rises control regulator valve **90** begins to open and drain condensate from the condenser **76** into the lower end of the heat exchanger coil **48**. Generally, make up water is not required because sufficient coolant fluid is provided by condensate from the steam used for initially heating up the oil and, accordingly, the valve **97** is generally closed. As heat is generated by the hydrogenation process, it is absorbed by vaporization of the coolant in the coil **48**. As the coolant vaporizes, more liquid coolant is introduced into the lower end of the coil until a state of equilibrium exists wherein the heat from the exothermic chemical reaction taking place in the vessel is absorbed by vaporizing the coolant, which vapor passes out of the vessel through the vapor line **54** into the entrainment separator **58**. The dry vapor entering the condenser is cooled by contact with the U-tubes **84** and is condensed into liquid form. Entrainment separator **58** reduces the amount of liquid entering the condenser shell, and this small amount of condensate is recirculated and returned to the coil directly via the return line **68**.

Heat generated by the chemical reaction taking place in the vessel **10** is removed at a controlled rate by vaporizing the coolant fluid in the coil **48** to maintain a desired oil temperature. This heat is then removed from the recirculation system in the condenser **76** by exposed tubes carrying the condenser coolant water. The temperature controlled regulator valve **90** regulates the flow rate of condensate from the condenser **76** in a precisely controlled amount to exactly expose sufficient condenser tube area to balance the amount of heat generated by the exothermic reaction of the hydrogenation process.

Because the recirculation system for the coolant fluid is closed, the pressure in the coil and condenser and other portions of the system can be above or below atmospheric and, accordingly, the varporizing temperature of the coolant vaporizing in the coil **48** is not fixed as in atmospherically vented systems but varies with the pressure. If the desired hydrogenation or reaction temperature is to be 350° F., for example, the present invention, using the closed condensation cooling system, can be set up to control and maintain this temperature within plus or minus 3° F., even though water is the coolant used. When hydrogenation is complete, the batch of oil is cooled to a much lower filtering temperature, such as 180° F. The temperature regulating valve **92** is set to a lower control point which causes the valve to go completely open and drain all the condensate from the condenser **76**, thus exposing all of the condenser surface. This causes the condensing system pressure to decrease until it nearly corresponds to the cooling water temperature. When the water enters via line **80** at 80° F. and exits via line **82** at 100° F., the pressure in the condensing system is about 1 p.s.i. absolute. It is to be understood, of course, that other types of heat exchange media can be used and that the invention is not limited to the use of water.

From the foregoing, it will be seen that difficulties of the prior art systems have been eliminated in the new and improved method and apparatus of the present invention. The coolant fluid can be maintained at an operating temperature range much closer to the desired oil temperature than hereto possible so that when desired heat may be removed from the oil without substantially changing its temperature. Moreover, violent flash-off and resulting vibration and hammering in the heat exchanger coil are eliminated. Precise control over the operating temperature is achieved and a high degree of controlled repeatability is possible with the method and apparatus of the present invention.

Although the present invention has been described by reference to only a single embodiment thereof, it will be apparent that numerous modifications and embodiments may be devised by those skilled in the art.

For example, the heat exchanger coil **48** may be replaced or supplemented by a jacket on the vessel **10**. In some installations vapor transferred from the heat exchanger coil **48** to the condenser **76** may contain so little liquid that the entrainment separator **58**, may be unnecessary. Condensers other than the U-tube type may suitably be employed.

When the hydrogenation process is practiced in equipment in which the heat exchanger holds a quantity of coolant which is large relative to the heat generated by the reaction, recirculation of coolant is unnecessary, and the reaction temperature can be controlled by draining condensate from the condenser and merely draining it to waste.

The apparatus and method of this invention may be employed for other exothermic reactions, and it is intended by the appended claims to cover all modifications and embodiments which will fall within the true spirit and scope of the present invention.

What is claimed as new and desired to be secured by Letters Patent of the United States is:

1. A method of batch hydrogenation of unsaturated oil according to a time-temperature schedule comprising the steps of heating the oil by condensing a heat exchange medium under pressure to provide a body of liquid medium in thermal communication with said oil, introducing hydrogen into the heated oil to establish an exothermic hydrogenation reaction, reducing the pressure over said medium to vaporize a portion of said medium and thereby adjust the temperature to the value scheduled for the hydrogenating oil and, subsequently, further reducing the pressure over said liquid medium to cool the oil to a filtering temperature.

2. The method of claim **1** wherein the hydrogenation temperature is from about 240° F. to about 450° F., the heat exchange medium is water and the vaporization takes place at an absolute pressure of from about 25 p.s.i. to about 425 p.s.i.

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