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Specs for a Bench-Scale Reaction Vessel and Cooker

This versatile unit helps to solve some of the problems of difficult-to-handle materials

Some plasticlike materials, slurries, solids, or semisolids are difficult or impossible to handle quantitatively in conventional laboratory equipment. Such a problem was encountered with flaked cottonseed meats, a material which, at certain conditions of heating and moisture content, forms a tough plastic mass. A cooker unit, designed to handle this problem, is particularly useful for process reactions or treatments requiring thorough mixing and kneading of materials at temperatures ranging up to about 170° C., with pressures of not over about 5 p.s.i. It's also useful as a general purpose reaction vessel by varying the type of agitator used.

Design of the unit enables quantitative recovery of all material components with nearly analytical accuracy, permitting precise material balances. Temperature, rate of addition, and time of retention of volatile liquids can be closely controlled.

Construction

The unit (Figure 1) consists of the jacketed vessel and accessories. All parts that contact the material being processed are constructed of stainless steel.

The sides of the steam jacket, *A*, were fabricated of 6- and 8-inch diameter pipes with 0.28-inch wall thickness, and the top and bottom closures of 0.25-inch plate. This jacket was tested at 120 p.s.i. Calculations indicate that it will withstand internal pressure in excess of 200 p.s.i. It is equipped with $^5/_8$-inch steam, *S*, and condensate, *W*, lines, a pressure gage, and a relief valve. The steam and condensate lines are also suitably connected to a controlled-temperature water supply to enable operation at temperatures below 100° C.

A removable liner, *B*, consisting of a commercially available 3-liter stainless steel beaker, is closely fitted into the machined interior of the jacket and is secured against rotation by means of a small lug, *U*, welded to the liner. This lug engages a stud, *V*, which projects vertically from the top edge of the inner wall of the jacket. The lip of the liner projects slightly above the edge of the jacket to enable positive contact with the cover, *C*. The cover is secured in place by means of three lug bolts, *T*, which engage notches in the cover. A vaportight seal between the cover and the edge of the liner is provided by a compressible gasket, *D*, of *Neoprene or Teflon.*

The cover, of 0.187-inch plate, is provided with a removable mounted combination shaft bearing and gland, *E*, and agitator, *F;* a thermometer well, *G;* a spray nozzle, *H;* four nipples, *I;* and two stationary blades, *J*. The agitator and stationary blades were designed with strength, rigidity, and thorough mixing and kneading action as primary considerations.

The stainless steel shaft is $^5/_8$ inch in diameter and the agitator blades were fabricated of $^1/_2$-inch bar stock. The horizontal blade, welded to the shaft, was bent and ground to shape and terminates at each end in integral vertical blades $2^1/_4$ and 4 inches in length, respectively. Both horizontal and vertical blades were ground to present faces inclined at an angle of 30° to the direction of rotation, so as to sweep material away from the bottom and sides of the vessel. The vertical terminal blades were ground to a cross-sectional shape somewhat like the cross section of an airplane wing, with the thin edge leading to minimize adherence of material. A third vertical blade, $4^1/_2$ inches in length, was welded to the horizontal blade $1^1/_4$ inches from the center of the shaft. This blade was ground to a triangular shape, approximately $^1/_4$ inch across the base and $^5/_{16}$ inch along the sides, with the sharp edge leading. The agitator was contoured to fit the liner as closely as possible, but still permitting free rotation.

The two stationary blades, *J*, projecting vertically downward from the cover into the vessel, together with the thermometer well, *G*, counteract the tendency of plastic materials to rotate with the agitator, and in conjunction with the vertical blades of the agitator give an effective kneading and shearing action. These stationary blades are $6^7/_{16}$ inches long, $^5/_{16}$ inch wide, and $^1/_8$ inch thick, with the shape of a two-edged knife. They are adjustably mounted through the cover and secured in place by lock nuts. The thermometer well is of thin-walled $^1/_4$-inch tubing and is sufficiently rigid to withstand bending with any material processed to date. Intimate contact of this well with the material being processed permits accurate temperature readings with either a mercury-glass thermometer or a thermocouple without appreciable lag when a few drops of liquid, such as glycerol, are used to facilitate heat transfer. The spray nozzle, *H*, screws onto the end of a $^1/_4$-inch pipe which is threaded through the cover and is fitted on its external end with a small needle valve, *K*. The needle valve is connected by a pipe union to the vertically mounted tube, *L*, which in turn is connected to a source of compressed air or gas. Tube *L* serves as the container for liquid to be added. The heavy-walled glass tube, *R*, inserted in the line enables visual determination of the instant when all liquid has been discharged from *L*. The four threaded nipples, *I* ($^1/_2 \times 1^1/_4$ inches), are welded through the cover and, when not in use, can be closed with threaded caps.

The agitator driving mechanism is mounted on a sturdy platform fabricated of $^1/_4$-inch steel plate and $1^1/_4$-inch angle iron. The drive comprises a $^1/_3$-hp. motor, *M*, flexibly coupled with a variable speed gear reducer, *N*, which is geared to a rigidly mounted vertical agitator drive shaft, *O*. Connection of the vertical drive shaft to the agitator shaft is by a standard flexible coupling, *X*. The rear support legs of the platform framework are attached by hinges, *P*, to the table top. The drive mechanism can be simultaneously disconnected from the stirrer at the coupling, *X*, and the cooker made fully accessible simply by swinging the assembly up and back on these hinges.

Operation

For quantitative work, materials may be weighed directly in the liner at the beginning and end of an experiment, and gain or loss, as from moisture changes, determined without sampling. This feature is particularly useful in processing oilseed materials, where changes in moisture content during processing are important and where it is extremely difficult to obtain representative samples from such relatively heterogeneous materials. In addition, oil-bearing materials tend to lose some oil by adherence to the walls and sides of processing vessels, and this loss is reflected in the quantity of oil found in samples of the material. By use of the liner the total quantity of material processed may be transferred directly to the extraction apparatus with no loss of oil or other componets.

To facilitate heat transfer between the jacket and liner, about 50 cc. of some high-boiling liquid, preferably glycerol, may be placed in the heating jacket before inserting the liner. Glycerol adhering to the liner on its removal from the jacket may be removed completely by wiping with a slightly damp cloth.

Water (or other liquids) may be metered into the vessel as a fine spray in the quantity and at the rate desired by proper manipulation of the needle valve in the calibrated spray nozzle. A measured amount of liquid contained in the vertical tube, *L*, is put under the pressure required for proper operation of the spray nozzle by means of compressed air or inert gas such as nitrogen. Addition of moisture at an even rate in the form of a fine spray has been found to be essential in obtaining an even distribution of moisture throughout the oilseed materials. Moisture or volatiles may be removed from materials being processed by passing a stream of air or inert gas over the material by means of the four nipple openings provided in the cover. Air or gas throughput may be metered with a gas rotameter. Volatiles may be discharged, collected, and measured by means such as condensers, absorption towers, or other collecting devices.

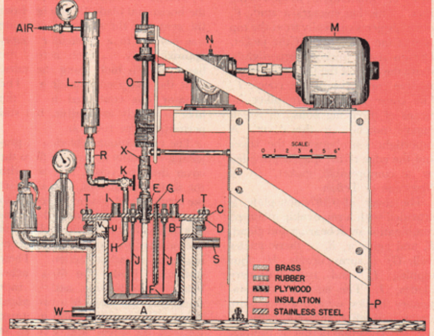

Figure 1. Bench-scale reaction vessel and cooker

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In the Equipment and Design Workbook Feature for August (p. 47 A), J. A. Kapnicky's address was incorrectly given as Monsanto Chemical Co. His correct address is Department of Chemical Engineering, University of West Virginia, Morgantown, W. Va.