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E. KÖNIGSTEIN

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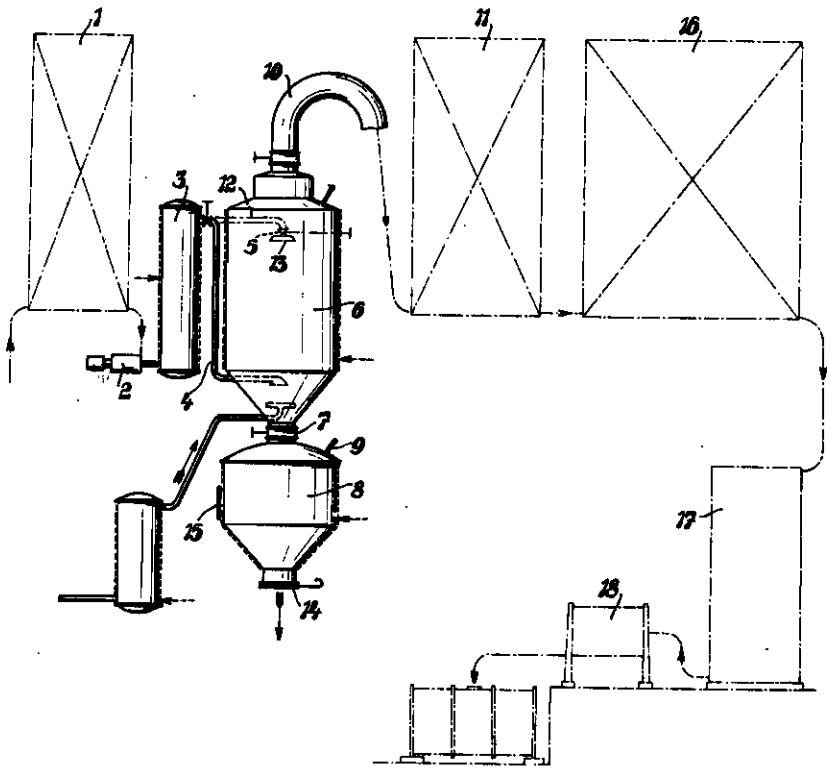

Inventor
E. Königstein

By: Glascock Downing & Seibold
Attys.

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PROCESS AND APPARATUS FOR OBTAINING GLYCERINE FROM DISTILLERY SLOPS OR SPENT WASH

Ernest Königstein, Malacky, Slovakia; vested in the Alien Property Custodian

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The slops or spent wash arising in the industrial production of spirit by fermenting crude sugar and sugar-containing raw substances, more particularly bect, contains a not inconsiderable quantity of glycerine. The processes heretofore proposed for obtaining this glycerine are satisfactory neither technically nor economically since they necessitate amongst other things extensive purification, not only physical but chemical as well, and clarification, of the spent wash being worked up, these operations having to be carried out in costly and expensive plants.

The present invention enables the glycerine to be obtained more economically and more simply technically from the slops or spent wash from distilleries even without any previous clarification and chemical purification. The process according to the invention consists essentially in this that the spent wash or slops arising from the distillery, merely neutralised and thickened, preferably not purified and not clarified, but advantageously heated under pressure to a temperature lying above its boiling point at atmospheric pressure, is not subjected in itself to distillation to crude glycerine, but is brought together in distillation spaces with distillation residues originating from the preceding working stage the temperature of which is equal to or higher than the distillation temperature of the orude glycerine to be distilled out at the working conditions coming into question. If the crude glycerine obtained thus according to the invention is freed from its organic impurities, it can then be directly further worked up by the usual methods to glycerine for any desired purpose of use.

The union of fresh slops and distillation residue in the distilling space, essential for the direct and smooth distilling out according to the invention, can be effected either by sucking the freshly supplied slops into the distillation residue and mixing up therewith or by introducing the previously suitably pressure heated slops in as fine a state of sub-division as possible into the preferably evacuated distilling space on to the surface of the distillation residue remaining therein, e. g. by causing the slops to sink into the surface layer of the residue by spraying on. The fresh slops introduced into the distilling chamber, preferably possess a temperature lying at least 20° above the boiling point thereof at atmospheric pressure, and possess also a super-atmospheric pressure of 2–3 atm. according to the density thereof and so forth.

The distillation effected under these conditions provided by the invention and also in the usual manner, e. g. by leading hot steam through the heated distilling chamber, as well as the subsequent condensation of the crude glycerine may be effected with continuous or intermittent withdrawal of the distillation residue collecting in the distilling chamber. In the case of intermittent withdrawal it has been found to be advantageous to withdraw from the vessel or heater on each occasion about one half of the residue remaining from the preceding charge of the distilling vessel and to make up the other half remaining in this to the original amount of charge by means of freshly supplied slops. In the case of uninterrupted withdrawal of the distillation residue the empirically established most favourable amount of charge is continuously maintained by likewise uninterrupted supply of a suitable quantity of fresh slops. In both cases the condensation of the crude glycerine obtained and usually also its distilling out from the slops can be conducted without interruption.

In order to obtain the first distillation residue necessary for starting the continuous distilling process and to bring it according to the invention to at least the distillation temperature of crude glycerine, there are expelled from an initial charge of the suitably pre-heated distillation vessel by any desired further heating thereof. first the water and readily volatile non-glycerine constituents and thereupon the glycerine, then the distillation residue so obtained is reduced to one half by withdrawal, and may be by continued heating brought to at least the distillation temperature of crude glycerine at the pressure prevailing in the vessel. Only now begins the afore-mentioned cnotinuous distillation operation effected by means of the blown-through steam by gradually running in fresh slops up to the point of re-attainment of the normal filling of the vessel.

The crude glycerine distilled out and condensed in known manner is, according to the invention, heated before its further working up to a temperature lying between its boiling point and that of water, that is to say for example between 100 and 140°, thereby held by continuously controlled additions of alkalising agents to an alkalinity of 0.8–1%, and after their stabilisation the decomposition products of the organic impurities are expelled by means of advantageously inert gases, such as for example carbon dioxide, or by means of super-heated steam being blown through.

The glycerine obtained thus can then be adapted in usual manner, e. g. by re-distillation, bleaching and deodourising, to any purpose of use.

The plant for carrying out the process comprises besides known neutralising, thickening, condensing, heating devices and so forth, also, according to the invention a distilling vessel the lower part of which is in direct connection by means of a wide regulably closable opening with a vessel for accommodating the fraction of the distillation residue to be drawn off which is capable of being evacuated and heated. In the

fresh slops supply lead, which either opens out in the lower part of the preferably evacuable distilling vessel or terminates in its upper part preferably with a distributing, injecting or spray device, a compressor is installed as well as a pre-heater for the slops being supplied. In order to influence the rapid expansion aimed at of the slops entering into the vessel under pressure, a pressure reducing valve capable of actuation from the exterior is advantageously installed directly in front of the outlet for the slops or before the distributing, injecting or spray device.

Example

The slops coming from the distillery column are neutralised by addition of lime and soda lye, thickened to a density of about 80° Balling in a vaporising plant 1 known per se, for example such as is indicated schematically in the drawing, at the same time heated to about 90° C., compressed by means of the pump 2 to a superatmospheric pressure of about 2 atms., forced through the steam-heated preheater 3, which it leaves with a temperature of 140–150° in order to enter, by way of pipe 4, into the lower part of the distilling vessel 6 which in the meantime has been heated to about 200° and evacuated. As soon as the slops have filled about one third of the distillation vessel, the further slops supply is interrupted, and by means of continued exterior heating with simultaneous enlargement of the vacuum in water contained in the slops together with the oils which may be present and other readily volatile organic constituents permitted to distil off. As soon as the temperature of the slops has arisen again to about 140–150° the crude glycerine is distilled out by introducing superheated steam, by opening the wide withdrawal opening 7 in the bottom of the vessel, after evacuation of the withdrawal container vessel 8 by means of its vacuum connection 9, about half of the residue remaining in the vessel is withdrawn into the said container, the withdrawal opening closed again and by renewed opening of the slops lead the vessel charge is gradually made up to the original amount. The distillation proper commences on introducing steam, which in contradistinction to the previously known method, takes place prefectly smoothly and quite rapidly amongst other things because of the avoidance of heat loss of the slops entering into the distilling vessel.

According to the prevailing working conditions, the pressure heated slops may also be supplied through the lead 12 as well as through the pressure reducing valve 5 and under sudden expansion be sprayed or ejected by means of the nozzle or sprinkler 13 on to the distillation residue remaining in the vessel, when the water and readily volatile constituents in the slops are to a great extent already distilled during the descent of the drops of slops and carried by the distilling steam into the condensing column 11.

In both cases the distilling vessel charge may be continuously maintained by controlled supply of fresh slops and corresponding withdrawal of distillation residue, and consequently also the distillation operation may be continuously maintained.

If the last traces of the glycerine contained in the slops are also to be obtained, the supply of slops is interrupted after suitable filling of the vessel, its distillate withdrawal lead 10 as well as its vacuum connection closed after distilling off the whole of the glycerine, about half of the distillation residue remaining in the vessel allowed to run off into the vessel 8, evacuated in the meantime, through the suitably opened withdrawal opening 7, the latter opening 7 closed again, the distillate withdrawal lead and the vacuum connection of the vessel opened, further slops supplied until the original degree of filling is attained, and the distillation further continued. This manner of working, even under difficult working conditions, makes possible the maintenance of the distillation vessel perfectly clean and the maintenance of a uniform degree of liquidity of the distillation residue and thereby its ready and rapid withdrawal. The condensation of the crude glycerine distilled off in this case also proceeds smoothly and without disturbance.

In order maybe to separate off more readily certain impurities otherwise distilling off together with the crude glycerine, then finally, although with abandonment of continuous operation, each vessel filling may also be completely distilled out itself and a commencement made with the new charging of the distilling vessel only after withdrawal of the whole of its distillation residue.

The emptying of the withdrawal vessel 8 takes place either by allowing the residue to flow out of the bottom valve 14 or by scooping it out through the lateral particularly wide withdrawal opening 15, according to the degree of liquidity of the distillation residue accumulated in the vessel 6.

In order to avoid corrosion, the inner walls of the distillation apparatus coming into contact with slops and crude glycerine are advantageously of copper, an iron-poor or iron-free alloy, or tinned.

The crude glycerine coming with a concentration of 85–90% from the condensation plate 11 is heated to above 100° before further working up. Average specimens are then withdrawn in brief intervals and corresponding to their acidity, determined in known manner, a quantity of milk of lime and sodium lye is added to the crude glycerine each time which just suffices to adjust its alkalinity to 0.8–1% and not to allow it to fall below 0.7%. This alkalisation is continued until the alkalinity has stabilised. Thereupon the volatile constituents and decomposition products of the organic, namely nitrogen-containing impurities contained in the crude glycerine are blown out by means of carbon dioxide or superheated steam and preferably the acidity of the glycerine obtained thus is adjusted to a pH value of 8 to 8.5.

According to the purpose of use in view if necessary the glycerine is thereupon re-distilled in a raffination plant 16 known per se, treated with active carbon in the apparatus 17 for the purpose of decolorisation or deodorising, forced through the filter press 18 and passed to conveying vessels as a product ready for use.

The utilisation of the hot vapour of the thickening plant, of the distilling and of the waste heating vapour in other stages of the total treatment, namely for feeding the pre-heater and so forth, is of course effected in accordance with the known principles of heat economy.

ERNEST KÖNIGSTEIN.