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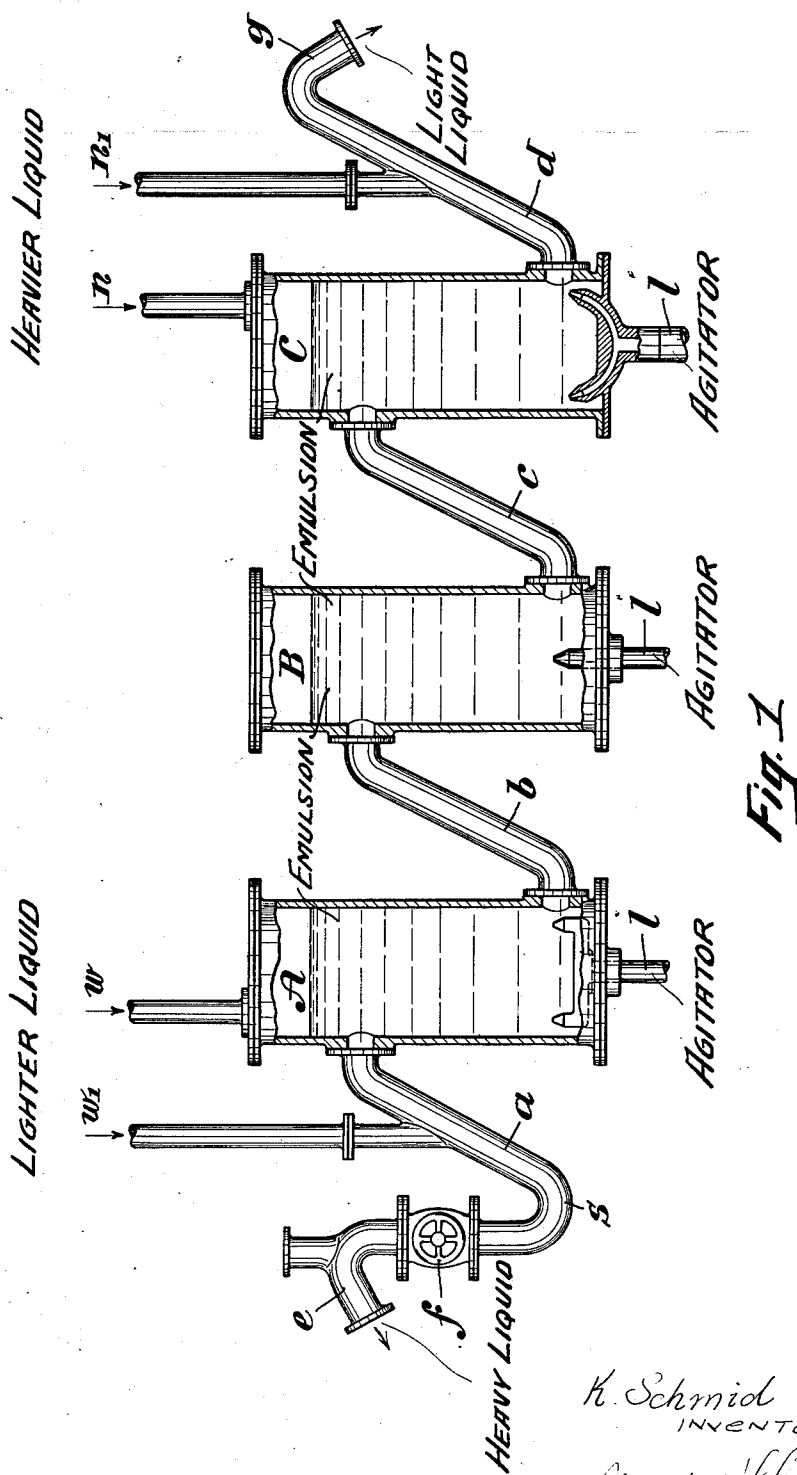
K. SCHMID

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PROCESS FOR THE CONTINUOUS MUTUAL REACTION OF TWO LIQUIDS OF DIFFERENT
SPECIFIC GRAVITIES ON THE COUNTER CURRENT PRINCIPLE

Filed Jan. 16, 1933

2 Sheets-Sheet 1



K. Schmid
INVENTOR
By: Marks & Clerk
ATTYS.

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K. SCHMID

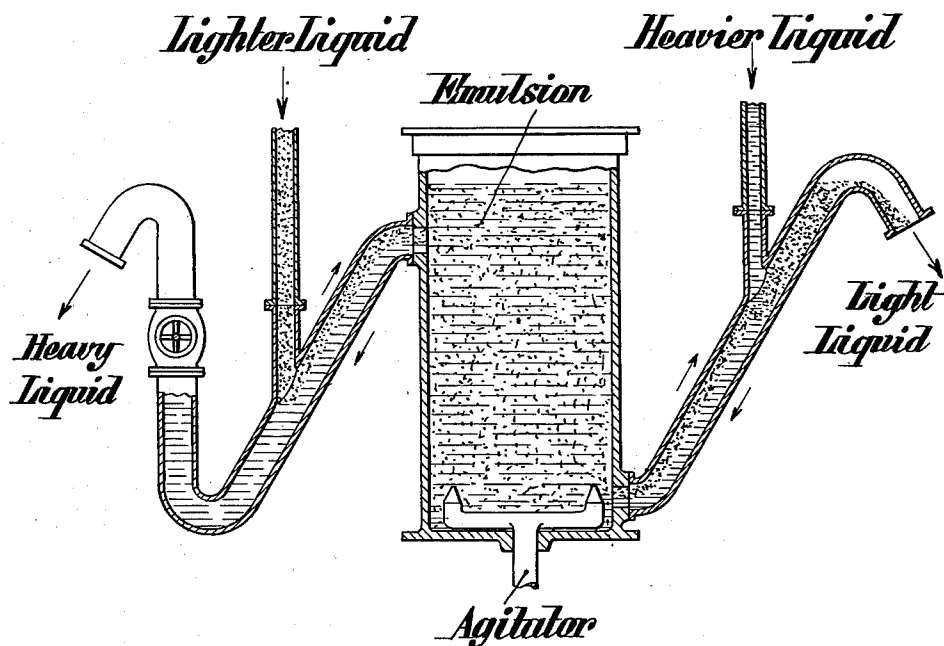
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2 Sheets-Sheet 2

Fig. 2



K. Schmid
INVENTOR

By: Marks & Clark Attys.

UNITED STATES PATENT OFFICE

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PROCESS FOR THE CONTINUOUS MUTUAL
REACTION OF TWO LIQUIDS OF DIFFER-
ENT SPECIFIC GRAVITIES ON THE COUN-
TER-CURRENT PRINCIPLE

Karl Schmid, Vaduz, Liechtenstein

Application January 16, 1933, Serial No. 652,094
In Germany February 12, 1932

1 Claim. (Cl. 23—270)

This invention relates to a process for the continuous mutual reaction of two liquids of different specific gravities on the counter-current principle in an apparatus composed of a number of mixers through which flow both liquids but in opposite directions with respect to one another. Communication between the mixers is established by one or more pipes ascending in the direction of flow, the first mixer and the last mixer being in communication with overflow pipes by means of an ascending pipe. According to this process, the heavier liquid is introduced into the mixer provided with the outlet for the lighter liquid, while the lighter liquid is admitted to the mixer at the opposite end of the apparatus. In view of this novel arrangement of the ascending communication pipes between the mixers it is possible to fully dispense with separators necessary when working according to known processes of continuous reaction on the counter-current principle. In these known processes, apparatus are employed in which mixing compartments are connected in series, however in these known apparatus a separator is arranged behind each mixing compartment in which the emulsion is separated, and thereafter the two liquids are again supplied to the next mixing compartment on the counter-current principle. Thus in the known processes a separator must be provided for every mixing compartment. A thorough separation of the liquids must take place in the separators, because otherwise the advantage of the counter-current principle is lost or otherwise diminished. In the known processes, the size of the separators must be suited to the speed of separation in order to cause a thorough separation. For instance in the case of washing nitroglycerine, the capacity of the separator must be suited for a flow of at least three quarters of an hour, for instance for a yield of 500 kilogrammes nitroglycerine per hour and a washing liquid of 500 kilogrammes, the separator must have a capacity of 750 kilogrammes. Now if six mixers are necessary for carrying out the process, the capacity of the separators has to be adapted to 4500 kilogrammes.

These drawbacks are avoided according to the present invention by arranging inclined passages between the mixers in place of separators.

Compared with the known example above described, in the event of like conditions, it is rendered possible according to the present invention to reduce the capacity of the communication passages between two mixing compartments to approximately 30 kilogrammes. In this case

all the communication passages together are of a capacity of no more than 180 kilogrammes. This advantage would be still further enhanced in the case of a process in which the separation is carried out still slower.

With respect to the solubility of the two liquids it is remarked, that the specifically heavier initial product must be difficultly soluble or quite insoluble in the specifically lighter final product, and the specifically lighter initial product must be difficultly soluble or quite insoluble in the specifically heavier final product.

The apparatus according to the invention is suited for the reaction of two convenient liquids of different specific gravities, such as for instance nitro-esters or nitro-products with water or alkaline solutions for their clarification or neutralization, benzol, hydro-carbons or other substances with acids for nitration, crude petroleum with acids for refining and the like.

Diagrammatic views of the apparatus according to the present invention are illustrated by way of example in Figs. 1 and 2 of the accompanying drawings, it being supposed that acid nitroglycerine is washed with an alkaline washing liquid for the purpose of neutralization.

Fig. 1 shows an elevation partly in section of a plant comprising three vessels.

Fig. 2 shows in sectional elevation a plant in which the dots represent the lighter liquid and the dashes indicate the heavier liquid.

In the drawings, A, B and C designate vessels which are open at the top and in which are mixed two liquids of different specific gravities for instance acid nitroglycerine and alkaline washing liquid.

For carrying out the mixing operation, air is blown through the tubes *l* and into the liquid. If desired also mechanical agitators, not shown in the drawings, may be arranged in the vessels. The vessels A, B and C are interconnected by ascending pipes *b* and *c*, while inclined pipes *a* and *d*, provided with overflow pipes *e* and *g* respectively, are secured to the outer vessels A and C respectively. A cock *f* serves for closing or opening the overflow pipe *e*.

Before the process is started, the entire apparatus is charged with washing liquid up to a level with the overflow pipe *g*. Agitation takes place within the vessels A, B and C. The cock *f* is shut. Washing liquid is continuously admitted by way of the inlets *w* or *w'* and flows uninterruptedly through the apparatus and finally leaves the same by way of the pipe *g*. The acid nitroglycerine uninterruptedly enters by way of

the inlets n or n' and, being the specific heavier liquid, flows through the pipe d and into mixer C. In view of the mixing an emulsion is formed in the mixer C and the acidity of the nitro-
 5 glycerine and of the washing liquid are balanced in accordance with the affinity of these two liquids with respect to the acid. The speed of the flow through the pipes a , b , c , and d can be controlled by suitably dimensioning the communication pipes. The emulsion in the mixer C
 10 is, however specifically heavier than the ascending lighter liquid for instance fresh water, and therefore the emulsion descends towards the ascending water in the direction towards the mixer
 15 B. The separators, which heretofore were arranged in apparatus operating on the counter-current principle between each pair of mixers, separated water and nitroglycerine according to the specific gravities. According to the present
 20 invention, a separation of both liquids is not necessary in view of the arrangement of the communication pipes. The emulsion in the mixer C consists of already washed nitroglycerine and acid water in fine distribution, whereby the nitro-
 25 glycerine so to say is in suspension in the acid water. Now if this emulsion sinks in the communication pipe c towards the ascending fresh water, the acid from the water-phase of the emulsion coming from the mixer C is washed back into the latter by the fresh water. Drops of nitro-
 30 glycerine which may pass into the mixer B are not separated, but the water surrounding the same does not contain any acid anymore, derived from the water-phase of the mixer C. The nitro-
 35 glycerine, which has been washed already, is thoroughly mixed again with fresh or weak alkaline water and passes in the same way through the communication pipe b and into the mixer A and from here through the pipe a into s . The nitroglycerine is completely separated from
 40 the water at s and leaves the apparatus by way of the outlet e whenever the cock f is opened and if in the pipe a a sufficient quantity of nitroglycerine has accumulated, so that at least the
 45 whole ascending pipe is filled between e and s , whereby this liquid-column of nitroglycerine prevents the discharge of water through this overflow. The height of the overflow pipe e
 50 must be adjusted in accordance with the specific gravities of both liquids and the now purified nitroglycerine flows out continuously by way of the pipe e . The washing water flows in a similar way towards the pipe g and leaves through the latter; so that the two liquids flow in opposite
 55 directions, i. e. in the counter-current principle.

In the vessel C, the washing liquid with the highest percentage of acid meets the nitroglycerine with the highest percentage of acid from which it still carries off some acid, while in the
 60 vessel A already cleaned nitroglycerine and clean washing liquid which may be slightly alkaline are meeting, whereby the latter fully cleans the former. Any desired number of mixing vessels
 65 A, B, C may be provided. In this way purified neutral nitroglycerine is obtained with the smallest quantities of water and alkali. An excess of alkali, taken in by the nitroglycerine during the washing process, can be washed out again in the
 70 vessel A, because clean water can be charged

into the vessel A while alkali is added in the vessel B only. Further it is possible to introduce or lead off any heat by enclosing the mixing vessels and separation pipes in a heating jacket or in a cooling jacket. Thus in this apparatus any separation of the two liquids according to their specific gravities does not take place between the mixing places, but practically the same effect can be attained in that the like phases assimilate and force back one another in the communication pipes through which the two liquids must flow in opposite directions to each other, because no other way of communication is at disposal. In the illustrated example, the acid from the water of the emulsion originating in the mixer C is washed back from the pipe c into the mixer C by the ascending fresh water.

The assimilation of like phases in emulsions is a general rule and therefore this is possible in all those cases in which two liquids can be washed, cleaned or enter into reaction on the counter-current principle.

By bending the communication pipes or by arranging obstacles, screens or the like within the same it is possible to readily mix both liquids during their passage in opposite directions through the communication pipes, whereby the assimilation to the like phases is promoted.

The construction of the mixers does not form a part of the present invention and therefore the same are illustrated diagrammatically in the drawing. The mixing is carried out by air current or by propeller but it must be such that the heavier liquid entering at the bottom is raised and is discharged at the top. If the overflow pipe e is disposed higher than the overflow pipe g , the apparatus will be filled with the heavier liquid and in this event the lighter liquid is forced downward by agitation from the top where it enters the mixer and leaves the same at the bottom outlet. The mixer may be such as is shown and described for instance, in the specification of U. S. Patent No. 1,893,447, dated January 3, 1933.

What I claim is:—

Process for the continuous mutual reaction of two mutually insoluble or difficultly soluble liquids of different specific gravities on the counter-current principle in an apparatus comprising interconnected mixers, which consists in introducing the lighter liquid at one end and the heavier liquid at the other end of the apparatus, allowing said liquids to flow continuously but in opposite directions through a passageway connecting two adjacent mixers and ascending in the direction of flow of the lighter liquid, whereby the heavier liquid descends through the ascending lighter liquid and washes back into the previous mixer from said lighter liquid the heavier particles carried by it and vice versa the lighter liquid ascending through the descending heavier liquid and washes back into the next mixer from said heavier liquid the lighter particles carried by it, and after leaving the first mixer and the last mixer allowing said liquids to ascend and descend respectively through passageways inclined in the same direction and connected with overflows.

KARL SCHMID. 70