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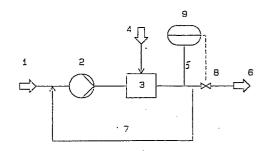
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(54) Equipment and process for dissolving gas in liquids.

The invention relates to equipment for dissolving gas in liquids, which equipment consists of an inflow (1), a delivery pump (2), a reaction zone (3), a gas feed line (4) leading into the reaction zone (3), a measuring probe (5) fitted downstream of the reaction zone (3), an outflow (6), a recycle line (7) through which a part of the gas/liquid mixture continuously flows back to a point unpstream of the reaction zone (3), and of a control element which is provided in the outflow (6) downstream of the recycle line (7) and which is opened and closed as a function of the control variable detected by the measuring probe and of a predetermined set value. The invention also relates to a process for dissolving gas in liquids by means of the equipment according to the invention.

<u>Fia.1</u>



Description

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Equipment and process for dissolving gas in liquids

The present invention relates to equipment and to a process for dissolving gas in liquids.

The state of the art has disclosed various types of equipment and processes for dissolving gas in liquids, in particular for the neutralization of alkaline liquids by means of carbon dioxide. Such units essentially consist of a delivery pump, a CO₂ mixing system, a reaction zone or a corresponding reaction vessel and a pH measuring point. Control is effected as a function of the CO₂ rate. From German Patent Specification 2,638,428, a process for neutralizing alkaline effluent by admixture of a neutralizing gas is known, wherein the effluent and a gas are fed separately to a nozzle of special design. Moreover, German Patent Specification 2,627,880 relates to a process for atomizing liquids by means of gases or for dividing gases into small bubbles by means of a liquid, the two phases being mixed with one another.

However, all the known processes for dissolving gases in liquids have disadvantages. Thus, these processes require expensive and large units and hence also high investment costs. A further point is that considerable expense on control is necessary, since high-value expensive control instruments must be used. There is also a risk of the gas not being completely dissolved, so that a disproportionately large part of the gas escapes. Likewise, the processes are unsuitable for elevated temperatures. Moreover, the known units can be used only for batch operation and not continuously. Finally, the units must be tailored to the specific application, so that standardization is impossible. The neutralization units according to the state of the art have the additional disadvantage that sharp alkalinity peaks cannot be neutralized. As a consequence, the pH value in the outflow fluctuates widely. At low pH values, the CO₂ conversion is poor, with the consequence of operation at reduced load due to escape of CO₂ gas.

It was therefore the object of the present invention to provide equipment for dissolving gas in liquids, which equipment consists of an inflow, a delivery pump, a reaction zone, a gas feed line leading into the reaction zone, a measuring probe fitted downstream of the reaction zone and an outflow, in order to overcome the disadvantages of the state of the art.

This object is achieved when a recycle line, through which a part stream of the reaction mixture continuously flows back to the suction side of the pump upstream of the reaction zone, is located downstream of the measuring probe and, in the outflow downstream of the recycle line, a control element is provided which is opened and closed as a function of the control variable detected by the measuring probe and of a predetermined set value. Depending on the position of the control element, a recycle rate/outflow rate ratio is established which is automatically adjusted to the extent of the reaction at the time.

The measuring probe is preferably an instrument, by means of which the pH, pO₂, pCO₂ and rH value can be determined. The reaction zone is one of the reactors usual according to the state of the art. For example, the nozzle shown in German Patent Specification 2,638,428 can be used. A buffer vessel with a device for controlling the liquid level and with an inflow can be provided upstream of the feed line. Any type of control elements can be used in the outflow. However, motorized control elements are particularly preferred.

It is a further object of the present invention to provide a process for dissolving gas in liquids by introducing a liquid and a gas into a reaction zone and discharging the gas-saturated liquid via an outflow.

This object is achieved when the liquid/gas mixture is continuously recycled via a recycle line to the liquid inflow, a control variable is detected by means of a measuring probe which is located between the reaction zone and the recycle line, and a control element in the outflow is opened or closed as a function of the control variable and of a predetermined set value.

For example, the pH, pO₂, pCO₂ and/or rH values can be determined by means of the measuring probe. The gases used are especially carbon dioxide, oxygen and hydrogen.

The process according to the invention is particularly applicable to the neutralization of liquids by means of carbon dioxide. The unit is very particularly suitable for the neutralization of strongly alkaline liquids. The pH value of the liquids fed in can be above 12. A particularly preferred range is a pH value between 12 and 14. The gas used for the neutralization is preferably carbon dioxide. As a result of the process according to the invention, it is possible to dispense with expensive reaction zones in a neutralization. Consequently, space-intensive and cost-intensive post-reaction zones are no longer necessary. In fact, by means of the recycle mechanism, the liquid can be circulated, and carbon dioxide can be added to it, until neutralization has taken place. The pH value is determined continuously by a measuring probe. When a certain pH value is reached, a valve in the outflow is opened by a control device, so that the neutralized liquid leaves the unit. The pH value of the liquid flowing out is between 6.5 and 9.0, preferably 8.

The process according to the invention and the equipment according to the invention substantially differ from the existing state of the art, in particular by the recycle of a part stream to the place of reaction and by the control depending on the flow rate. This is a novel process concept which avoids all the existing disadvantages of the state of the art. The result is a simple and inexpensive alternative to the existing state of the art.

The process can be universally employed wherever gases of low solubility, such as H₂ and O₂, are to be dissolved in a liquid almost up to the saturation limit. Moreover, the process is suitable for accomplishing chemical liquid/gas reactions at an optimum with regard to materials, engineering and economics. Finally, the process also allows gases to be stripped out of contaminated liquids.

Above all in the case of neutralization, the process according to the invention has considerable advantages

over the existing state of the art. By means of the process, it is possible that in fact only neutralized effluent is discharged, in accordance with the conditions imposed by the authorities. The neutralized water flowing out and the recycle water rate are automatically and dynamically adjusted in accordance with the extent of neutralization at the time. Furthermore, effluents having a pH value at any high level can be neutralized. This means that even very high alkalinity values, which the existing state of the art was unable to eliminate satisfactorily, are amenable to neutralization by means of the process according to the invention. Moreover, the unit according to the invention can be used for both continuous operation and batch operation. The gas rate/effluent rate ratio is always adjusted to a constant value. The consequence is that the reaction system always runs at the optimum operating point with the maximum efficiency. Especially when the process according to the invention is used, the nozzle according to German Patent Specification 2,638,428 can reach a hitherto unknown efficiency. The recycle also has the advantage that it is possible to neutralize even at elevated temperatures, at which neutralization has hitherto involved difficulties. In this case, the neutralization does not require a special reaction vessel.

In the table which follows, the advantages of the process according to the invention are illustrated by comparison with the state of the art, which operates without recycle:

Comparison between a process for neutralizing a liquid and the process according to the invention

•	-	
Criteria	State of the art	Process according to the invention
process principle	linear	circulation
complete neutrality	not ensured	ensured
safety against impermissible outflow	not ensured	ensured
effluent/feed fluctuations rate	sensiti v e	insensitive
concentration	sensitive	insensitive
rate and con- centration	sensitive	insensitive
CO ₂ feed	pH-dependent	constant
outflow pH	fluctuating	constant
effect of effluent temperature	large	small
effective loop	open pH-regulated	closed pH-controlled
CO ₂ conversion	fluctuating	optimum
gas/water ratio	not optimizable	optimizable
mass transfer	varying	optimum

Table continued

State of the art	Process according to the invention	5	
poor	good	10	
poor	good		
not possible	readily possible	15	
in more detail below with referen	once to the figures	20	
In Figure 1, the basic principle of the equipment according to the invention is shown. In Figure 2, a particularly preferred embodiment is illustrated, which is preferentially suitable for the neutralization of strongly alkaline liquids by means of carbon dioxide. In Figure 1, the liquid is fed to the unit via the inflow 1. The liquid is fed via the pump 2 to the reaction zone 3. The gas which is to be dissolved in the liquid is also fed to this reaction zone via a line 4. The liquid/gas mixture			
is continuously monitored by the measuring probe 5. Depending on the gas fed or on the reaction, this can be a pH probe, pO ₂ probe, pCO ₂ probe or rH probe. As a function of the values measured, the valve 8, which is actuated via the control device 9, in the outflow opens or closes. Depending on the position of the valve 8, a part stream of the reaction solution is recycled via the line 7 and re-introduced via the pump 2 into the reaction zone 3, where gas is added again via the line 4. The rate leaving the unit via the outflow 6 is the difference between feed and part stream. At the same time, the corresponding liquid rate again enters the unit via the inflow 1. A recycled part stream/outflow rate ratio is established automatically in accordance with the predetermined set value. The result of this is that liquid is discharged via the outflow 6 only in accordance with the predetermined set value. By a suitable choice of the recycle ratio, liquids can be enriched with gas up to almost their saturation limit.			
into the reaction zone via the lines 15 and 4. At the same time, the effluent is fed back to the reaction zone 3 via the recycle line 7. When the effluent then reaches the set desired value at the measuring point 5, the control valve 8 opens and a part of the effluent passes into the outflow. At the same time, an equal part of untreated effluent passes into the inflow 1 and, mixed with the recycle stream 7, enters the reaction zone 3. In the latter,			
e controller 11, the control eleme	ent 8 consistently corrects the outflow	50	
n liquids, consistina of		55	
intribute established to the contribute of the state of t	n more detail below with reference the equipment according to the strated, which is preferentially substituted. Which is preferentially substituted is also fed to this reaction zuring probe 5. Depending on the rH probe. As a function of the resulting of the rH probe outflow opens or closes. Depending on the rH probe with the line 7 and re-introche line 4. The rate leaving the usame time, the corresponding like low rate ratio is established and is is that liquid is discharged via the level control 15, the substitute is in the reaction zone 3 to the rably is in the region of pH = 6. The carbon dioxide feed valved at the same time, the effluence of the recycle stream 7, eded to the mixture. If the value are controller 11, the control element of the co	good The possible readily possible The equipment according to the invention is shown. In Figure 2, a strated, which is preferentially suitable for the neutralization of strongly oxide. The liquid is fed via the pump 2 to the reaction zone 3, quid is also fed to this reaction zone via a line 4. The liquid/gas mixture ring probe 5. Depending on the gas fed or on the reaction, this can be rH probe. As a function of the values measured, the valve 8, which is outflow opens or closes. Depending on the position of the valve 8, a cycled via the line 7 and re-introduced via the pump 2 into the reaction he line 4. The rate leaving the unit via the outflow 6 is the difference same time, the corresponding liquid rate again enters the unit via the low rate ratio is established automatically in accordance with the his is that liquid is discharged via the outflow 6 only in accordance with ble choice of the recycle ratio, liquids can be enriched with gas up to refed to a collecting tank 16. As soon as an upper level point has been on via the level control 15, the switching point 10, the controller 11 and uent via the reaction zone 3 to the pIH measuring point 5. As long as pH rably is in the region of pH = 6.5 - 9, are indicated, the control valve 8. The carbon dioxide feed valve 14 is opened, so that the gas can flow d4. At the same time, the effluent is fed back to the reaction zone 3 via an reaches the set desired value at the measuring point 5, the control passes into the outflow. At the same time, an equal part of untreated xed with the recycle stream 7, enters the reaction zone 3. In the latter, ded to the mixture. If the value now measured at the pH measuring e controller 11, the control element 8 consistently corrects the outflow recycle rate, so that only effluent which meets the measured set value	

a) an inflow (1), b) a delivery pump (2), c) a reaction zone (3), d) a gas feed line (4) leading into the reaction zone (3), e) a measuring probe (5) fitted downstream of the reaction zone (3) and 60 f) an outflow (6), characterized in that

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g) a recycle line (7), through which a part stream of the gas/liquid mixture continuously flows back to a point upstream of the reaction zone (3), is located downstream of the measuring probe (5) and,

- h) in the outflow (3) downstream of the recycle line (7), a control element (8) is provided which is opened and closed as a function of the control variable detected by the measuring probe and of a predetermined set value.
- 2. Equipment according to Claim 1, characterized in that the measuring probe is an instrument for determining the pH, pO₂, pCO₂ and/or rH values.
- 3. Equipment according to one of Claims 1 to 2, characterized in that a buffer vessel (16) with a device (15) for controlling the liquid level and with an inflow (17) is located upstream of the feed line.
- 4. Equipment according to one of Claims 1 to 3, characterized in that the control element (8) is a motorized control element.
- 5. Process for dissolving gas in a liquid by introducing a liquid and a gas into a reaction zone (3), characterized in that
 - a) a part stream of the reaction mixture is recycled continuously via a recycle line (7) to the suction side (2) of the pump,
 - b) a control variable is detected by a measuring probe (5) which is located between the reaction zone (3) and the recycle line (7), and
 - c) a control element (8) in the outflow (6) is opened or closed as a function of the control variable and of a predetermined set value, and
 - d) a recycle rate/outflow rate ratio is automatically established depending on the position of the control element.
- 6. Process according to Claim 5, characterized in that the pH, pO₂, pCO₂ and/or rH values are determined by means of the measuring probe (5).
- 7. Process according to one of Claims 5 or 6, characterized in that the recycle rate is controlled by the control element (8).
 - 8. Process according to one of Claims 5 to 7, characterized in that CO2, O2 or H2 is used as the gas.
- 9. Process according to one of Claims 5 to 8, characterized in that, for neutralizing a liquid, CO₂ is fed and, at a pH value of between 6.5 and 9.0, preferably of 8, the control element (8) is opened in order to allow outflow of neutralized liquid.
- 10. Process according to Claim 9, characterized in that alkaline liquids having a pH value between 10 and 14 are neutralized.

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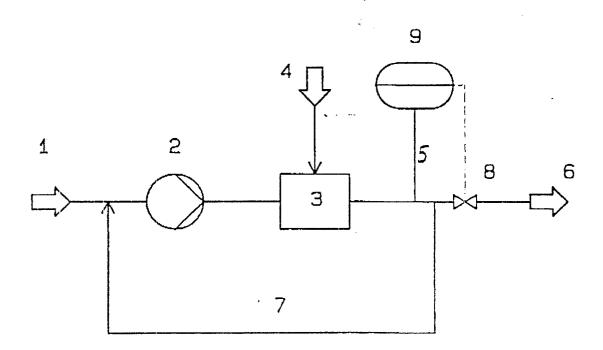
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