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PATENT SPECIFICATION

NO DRAWINGS

1,176,982

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Date of Application and filing Complete Specification: 1 March, 1967.

No. 9637/67.

Application made in United States of America (No. 532869) on 9 March, 1966.

Complete Specification Published: 7 Jan., 1970.

Index at acceptance: —B1 J(3A4, 3B1, 3B3, 3B4, 3CU, 4B, 4U, 6A2A, 6B3)

International Classification: —B 01 j 1/08

COMPLETE SPECIFICATION

Ion-Exchange Process

We, ROHM AND HAAS COMPANY, a corporation organized under the laws of the State of Delaware, United States of America, of Independence Mall West, Philadelphia, Pennsylvania 19105, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed to be particularly described in and by the following statement:—

This invention relates to a process for the removal of anions from a liquid containing them.

In accordance with the invention there is provided a process of the above type which comprises (1) passing the liquid downwards through a vessel containing an upper layer of weak base resin in the free base form and a lower layer of strong base resin in the hydroxide form, the weak base resin when in the free base form having an absolute density which is at least 0.01 g./ml. less than the absolute density of the strongly basic resin when in the hydroxide form and having a particle size relative to that of the strong base resin permitting hydraulic classification of the two resins, (2) backwashing the resins after the treatment cycle to remove loosely held impurities and air bubbles therefrom, the backwashing resulting in a degree of intermixing of the two resins, (3) regenerating the resins with a basic medium and (4) further backwashing the resins in the regenerated form to stratify them into an upper layer of the weak basic resin and a lower layer of the strong base resin. The weak base resin will contain, for example, groups of the formula $—N(R)_2$, i.e. in free base form. By contrast, a hydroxide form resin contains, for example, groups of the formula

$$—\overset{+}{N}(R)_3\overset{-}{O}H.$$

In the process of the invention, the upper layer of weakly basic resin acts as a scavenger of organic material and thus protects the lower layer of strongly basic resin and such upper layer also will remove strong acids leaving the weak acids to be removed by the strong base resin. The use of the two types of resins in the same vessel also results in a saving of equipment costs. Yet again the employment of stratified beds of resins results in much greater regeneration efficiency as compared with a single bed system in which the weak base and strong base resins are mixed together.

Typically the liquid to be treated by the process of the invention is water which has already had cations removed therefrom by treatment with a strongly acidic cation exchanger. Such a water will contain strong acids such as H_2SO_4, HCl and various organic acids which will be removed by the weak basic resin and weak acids such as H_2CO_3 and H_2SiO_3 which will be removed by the strong base resin.

In general, the particle size of the two types of resins will be from about twenty to about fifty mesh (U.S. Standard Sieve Series). For example the resins may have particle sizes such that all or a major proportion by weight thereof have sizes within the range of twenty to thirty mesh. With exchange materials that are carefully screened to obtain an even more uniform particle size, and particularly if the particle size of the more dense resin is greater than the particle size of the less dense resin, a very minimum difference in the densities will suffice. Of course, stratification of the resins is more readily accomplished as the differences in densities increase, and the same holds true if the particle sizes of the resins become more uniform. Desirably the two resins (when in the free base or hydroxide form as the case may be) have an absolute density

[*Price 5s. 0d.*]

difference which is at least 0.02 and better still 0.03 or 0.04 g./ml.

Resins which perform particularly well in the process of the invention are the weak base resin Amberlite IRA-93 which is a weak base macroreticular styrene-divinylbenzene copolymer carrying amine groups and the strong base resin Amberlite IRA-402 which is a strong base gel-type styrene-divinylbenzene copolymer carrying functional $-N-(CH_3)_3^+$ groups. The word "Amberlite" used above is a registered trade mark owned by the present Applicants.

The improvement in capacity which is accomplished by the backwash-resin stratification step can be appreciated from the following example of a typical application of the invention and the resulting data which are summarized in Table I.

EXAMPLE

Using a mixed bed in which the weakly basic resin was Amberlite IRA-93 and the strongly basic resin was Amberlite IR-402, a water sample was essentially exhausted. Following conventional practice, the bed then was backwashed with raw water (optionally, the purified effluent could have been used) to remove the impurities loosely held in the bed as well as air bubbles entrapped therein. This was followed by regeneration of the resins with caustic soda. The ion exchange capacity of this regenerated mixed bed was tested by exhausting it with another sample of water, the amount of that capacity being indicated as Operation A in Table I below. The thus-extracted resin was again backwashed to remove impurities and air bubbles, and regenerated again with caustic soda. This time, however, the regenerated bed was backwashed (once more) before testing the capacity of the resin mixture, the backwashing resulting in stratification of the resins. The capacity was then measured by exhausting the resin with another sample of water, the amount of that capacity being indicated as Operation B in Table I below.

Table I

	Ion Exchange Capacity in Kgrs. $CaCO_3$/ft.[a] Radio of Weak[(a)] to Strong[(b)] Base Resins		
Operation [(c)]	10/90	30/70	50/50
A	14	13	15
B	15	18	21

Notes:

(a) Weak base resin was Amberlite IRA-93

(b) Strong base resin was Amberlite IRA-402

(c) All data obtained after a 4 lbs./ft.[3] regeneration with caustic soda at 120° F. and with a water containing 90% strong acids and 10% weak acids.

It will be noted that in each instance where the regenerated mixed bed was given an additional backwashing step (Operation B) after regenerating but before using the resins, in accordance with the present invention, there was a marked improvement in capacity over the maximum capacity obtainable by the conventional practice of backwashing after resin exhaustion and then regenerating the resins and exhausting them again.

Clearly, stratification of the resins with the weakly basic resin on top is critical to the attainment of increased capacity, and this result can be readily obtained when the backwashing step is employed after the regeneration step. Not all weakly and strongly basic anion exchange resins, however, lend themselves to this technique. For example, when Amberlite IR-45, a weakly basic resin, and either Amberlite IRA-402 or Amberlite 900, both strongly basic resins, were employed, an entirely different and unsatisfactory result ensued. No stratification resulted when the regenerated mixture of either pair of these resins was backwashed, and absolutely no increase in capacity developed when either pair was submitted to capacity-exhausting measurements.

WHAT WE CLAIM IS:—

1. A process for the removal of anions from a liquid containing them which comprises (1) passing the liquid downwards through a vessel containing an upper layer of weak base resin in the free base form and a lower layer of strong base resin in the hydroxide form, the weak base resin when in the free base form having an absolute density which is at least 0.01 g./ml. less than the absolute density of the strongly basic resin when in the hydroxide form and having a particle size relative to that of the strong base resin permitting hydraulic classification of the two resins, (2) backwashing the resins after the treatment cycle to remove loosely held impurities and air bubbles therefrom, the backwashing resulting in a degree of intermixing of the two resins, (3) regenerat- the resins with a basic medium and (4) fur-

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ther backwashing the resins in the regenerated form to stratify them into an upper layer of the weak base resin and a lower layer of the strong base resin.

2. A process according to claim 1 wherein the particle size of the resins is from 20 to 30 mesh (U.S. Standard Sieve Series).

3. A process according to claims 1 or 2 wherein the liquid to be treated in water which has previously been treated with a strongly acidic cation exchanger to remove cations therefrom.

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Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1970.
Published by the Patent Office, 25, Southampton Buildings, London W.C.2, from which copies may be obtained.

