

Avoiding, removing or lessening deposits on apparatus parts in the esterification of acrylic or methacrylic acid

5 The present invention relates to a method of avoiding, removing or lessening deposits on the surfaces of apparatus parts in the preparation of esters of acrylic or methacrylic acid by esterification of the corresponding acid with an alcohol.

10 It is known that esters of acrylic or methacrylic acid can be prepared industrially by catalyzed esterification of acrylic or methacrylic acid with the corresponding alcohols. This often results in the formation of relatively high molecular weight by-
15 products which, inter alia, lead to deposits on the surfaces of the apparatuses used for the esterification reaction or downstream apparatuses or apparatus parts, eg. on the trays of a separation column. The deposits lead to a restriction in function or a malfunction of the apparatuses and thus to a deterioration
20 in the product quality of the reaction product, which makes cleaning of the corresponding apparatuses essential.

It has now been found that the corresponding deposits on the metal, glass, ceramic or plastic surfaces of the apparatuses or
25 apparatus parts can be avoided, removed or lessened if a liquid comprising the alcohol used for the esterification or consisting of this alcohol is allowed to act on the deposits or the surfaces of the apparatuses or apparatus parts on which such deposits have formed or are easily formed.

30 The present invention accordingly provides a method of avoiding, removing or lessening deposits on the surfaces of apparatus parts used for the esterification reaction and/or downstream apparatus parts in the preparation of esters of acrylic or methacrylic acid
35 by esterification of the corresponding acid with an alcohol having, in particular, from 1 to 4 carbon atoms, preferably in the presence of a strong acid such as sulfuric acid, p-toluene-sulfonic acid or acid ion exchangers, which comprises allowing a liquid comprising or consisting of the alcohol used for the esterification to act on the deposits or on the surfaces of the apparatus parts on which such deposits form. In general, the liquid
40 should comprise at least 10 and in particular at least 50 % by weight of the alcohol used for the esterification; it preferably consists essentially (entirely or virtually entirely)
45 of the alcohol used for the esterification. However, cleaning the deposits from the apparatus surface in this way or lessening or avoiding such deposits does not succeed when using the corres-

ponding ester formed in the esterification reaction, the acid used or water, but only when using the alcohol used in the esterification either alone or in combination with one or more of the above constituents.

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It has been found to be extremely advantageous to use a nozzle or a plurality of nozzles or sprayers by means of which the entire surface of the relevant apparatus parts on which deposits have formed or easily form can be wetted or sprayed with the alcohol-
10 containing liquid. This wetting by means of a nozzle or spraying of the apparatus surface can be carried out discontinuously or preferably continuously. It is thus possible to clean the surface of the relevant apparatuses or apparatus parts or to maintain a deposit-free surface so as to lead to a constantly high product
15 quality of the reaction product. Suitable nozzles or sprayers are commercially available nozzles and sprayers such as center body nozzles, rotating spray nozzles and also wide-area cleaning sprayers. Preference is given to nozzles which produce alcohol droplets having a droplet diameter of less than 1 mm on the
20 apparatus surface. It is noteworthy that the addition of a corresponding excess of alcohol to the esterification mixture produces no corresponding cleaning action.

25 Apparatuses or apparatus parts which have a tendency to suffer from deposits of relatively high molecular weight by-products are those which are used for the esterification reaction, but also the apparatuses and apparatus parts downstream of the actual esterification reaction, for example separation columns, which
30 come into contact with the esterification mixture and are fouled by deposits and as a result of this cause, for example, a pressure drop in the trays of the column.

The following example and the comparative experiments are to
35 illustrate the invention but do not restrict it in any way.

Example 1

40 The esterification of methacrylic acid with methanol in the presence of p-toluenesulfonic acid was carried out in a reaction vessel having natural convection. A separation column having 20 trays was superposed on the reactor. The distance between the liquid surface and the lowest tray was 2.2 m. The column diameter
45 was 0.8 m.

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A commercial tangential solid center body nozzle was installed 30 cm below the lowest tray. At a continuous liquid throughput of 100 kg/hour of methanol through the nozzle, the entire underside of the tray could be wetted in this way. The methanol droplets produced had a droplet diameter of from 20 μ m to 1 mm. The increase in the pressure drop over the lowest tray of the column was employed as the measure for the degree of fouling of the column. The pressure drop increased by 1 mbar over a period of 10 days and then remained constant.

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Comparative Experiment 1

The esterification reaction was carried out as in Example 1, but the nozzle was not installed. The corresponding amount of the methanol introduced through the nozzle in Example 1 was here added directly to the esterification mixture in the reactor. The pressure drop over the lowest tray of the column increased by 3 mbar over a period of 10 days and rose by a further 10 mbar over the course of the next 60 days.

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Comparative Experiment 2

The esterification reaction was carried out as in Example 1, but the nozzle was not installed. Unlike Comparative Experiment 1, the amount of methanol introduced through the nozzle in Example 1 was not introduced into the esterification mixture in the reactor either. The pressure drop over the lowest tray of the column increased by 3 mbar over 10 days and rose by a further 10 mbar over the course of the next 60 days.

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Comparative Experiment 3

The esterification reaction was carried out as in Example 1, but the apparatus parts were wetted by means of the nozzle using methyl methacrylate in place of methanol. The fouling of the separation column could not be avoided in this way. The pressure drop over the lowest tray of the column increased by 3 mbar over a period of 10 days and rose by a further 10 mbar over the course of the next 60 days.

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REPLACED

We claim:

- 5 1. A method of avoiding, removing or lessening deposits on the surfaces of apparatus parts in the preparation of esters of acrylic or methacrylic acid by esterification of the corresponding acid with an alcohol, which comprises allowing a liquid comprising or consisting of the alcohol used for the esterification to act on the deposits or the surfaces of the apparatus parts on which such deposits form.
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2. A method as claimed in claim 1, wherein the liquid allowed to act on the deposits or the surfaces of the apparatus parts comprises at least 10 % by weight of the alcohol used for the esterification.
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3. A method as claimed in claim 1 or 2, wherein the surfaces of the apparatus parts on which deposits have formed or on which deposits easily form are wetted with the alcohol-containing liquid by means of nozzles or cleaning sprayers.
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4. A method as claimed in any of claims 1 to 3, wherein the apparatus parts are parts of a separation column used for the esterification reaction or installed downstream of this.
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5. A method as claimed in any of claims 1 to 4, wherein, in the preparation of methyl methacrylate, methanol-containing liquids are allowed to act on the deposits or the surfaces of the apparatus parts.
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6. A method as claimed in any of claims 1 to 5, wherein the liquid allowed to act on the deposits or the surfaces of the apparatus parts consists essentially of the alcohol used for the esterification.
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7. A method as claimed in any of claims 1 to 6, wherein the liquid is allowed to act continuously on the surface of the corresponding apparatus parts.
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