

UNITED STATES PATENT OFFICE

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DESIZING AGENT

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5 Claims. (Cl. 195-64)

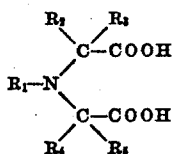
This invention relates to improvements in amylase preparations.

It is known practice to use for technical purposes, especially for desizing, amylases. These enzymes are very sensitive towards salts of heavy metals, particularly those of copper and zinc. Even the traces of such metal salts which frequently occur in town water supplies may considerably affect the action of an enzyme liquor prepared with the water. In the technical application of the amylases there arise considerable disturbances through heavy metals. Such disturbances are especially noteworthy when the amylases are used for removing sizes which contain copper or zinc salts, which is frequently the case since such metallic salts are often added to the sizing solution to avoid fermentation and putrefaction phenomena.

It has been proposed to add to the amylase preparations protective agents of various kinds. However, these agents do not afford complete protection for the amylases against poisoning by heavy metals. In some cases the activity of the protective agent is diminished by heat or the agent produces troublesome precipitations by the action of lime salts; in other cases good durable mixtures of the enzyme preparation with the protective agent cannot be attained.

According to the invention protection of amylases is attained in an especially advantageous manner by combining the amylase with imino-diacetic acid or a substitution product thereof (see French Patent 811,938). An object of the invention are amylase preparations comprising protective agents of the mentioned kind. Another object of the invention is a process of desizing wherein starch sized material is treated with amylase preparations comprising the said protecting agents.

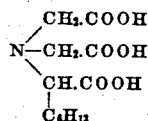
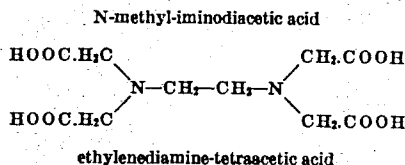
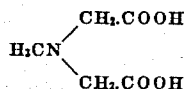
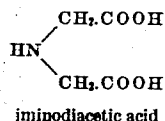
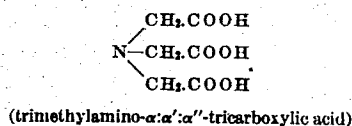
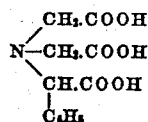
As substituents in the substitution products of the imino-diacetic acid the most important are aliphatic or cycloaliphatic or aromatic residues. These residues may, if desired, themselves contain further substituents, for example carboxylic acid groups. Such compounds correspond with the general formula:

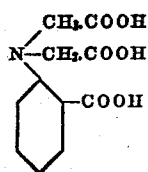


in which R_1 to R_5 stand for hydrogen, an al-

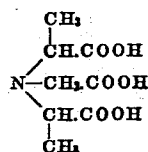
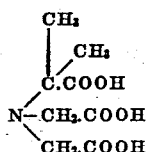
phatic or a cycloaliphatic residue or an aromatic residue. It is advantageous to use compounds in which merely the hydrogen atom attached to the nitrogen atom of the imino-diacetic acid is exchanged for a substituent.

Instead of the said acids themselves salts thereof may be used, but obviously only such salts of the imino-diacetic acid or its substitution products of which the cation does not itself have an injurious action on the amylase. Moreover, the salts used must be soluble in water. It is generally sufficient, however, if the salts are only slightly soluble in water, since the heavy metal compounds which are to be rendered harmless by the salts are present in the desizing liquor for the most part only in small proportions. There follows a list of compounds which may be used in the invention:

C-hexyl-trimethylamine- α,α',α'' -tricarboxylic acidC-phenyl-trimethylamine- α,α',α'' -tricarboxylic acid



N-(ortho-carboxy-phenyl) iminodiacetic acid

C, C'-dimethyl-trimethylamine- α , α' , α'' -tricarboxylic acidC, C-dimethyl-trimethylamine- α , α' , α'' -tricarboxylic acid

Moreover, there may be used for example diethylenetriaminoacetic acids which are obtained by the reaction of diethylenetriamine with chloroacetic acid.

The aforesaid compounds surpass to a considerable degree the protective agents hitherto added to enzyme preparations. They afford a practically complete protection also, which even remains when the temperature is raised. The compounds do not yield with lime salts any disturbing precipitation and are also effective in very hard water. They form, for example when used as their salts, mixtures with enzyme preparations that are good durable commercial products; unlike other protective agents they are equal in their effect towards different metals, for example zinc and copper. The protective action of the substances in question is available for amylases of any origin, for example in the case of pancreas amylases or bacterial amylases. A protective agent is preferably added, for example in the form of its alkali salt, to the amylase preparation during the manufacture of the latter. The enzyme preparation may also receive otherwise usual additions such as buffer substances, stabilising agents and the like.

The following examples illustrate the invention, the parts being by weight:

1. A desizing agent of very good effect, which is insensitive to the traces of copper and zinc present in the usual town water supply, is obtained by mixing 5 parts by weight of dry pancreas (Willstätter and Waldschmidt-Leitz, Hoppe-Seyler Zs. f. physiol. Chemie, Bd. 125, 150), 92 parts by weight of sodium chloride, 2 parts by weight of sodium salt of trimethylamine- α : α' : α'' -tricarboxylic acid and .8 part by weight of primary sodium phosphate.

2. 100 parts of cotton fabric carrying a starch size which contains zinc chloride are desized at 45° C. with 500 parts of a solution adjusted to pH=7 and containing per liter .1 gram of dry pancreas, 2 grams of sodium chloride and .2 grams of sodium salt of trimethylamine- α : α' : α'' -tricarboxylic acid. After one hour complete desizing has generally occurred.

3. A desizing agent which retains its activity

in the presence of impurities comprising heavy metal salts consists of 4.5 parts of dry amylase preparation made from *Bacillus mesentericus*, 46 parts of sodium chloride, 45.5 parts of anhydrous sodium sulphate, 1.5 parts of primary sodium phosphate and 2.5 parts of sodium ethylene diamine tetracetate.

4. A desizing agent is made by mixing 4 parts of dry pig pancreas, 2 parts of calcium formate, 86 parts of sodium chloride, 5 parts of anhydrous sodium sulphate and 3 parts of potassium salt of trimethylamine- α : α' : α'' -tricarboxylic acid.

5. A desizing agent consists of 6 parts of dry pig pancreas, 2 parts of primary sodium phosphate, 90 parts of sodium chloride and 4 parts of sodium iminodiacetate.

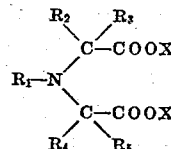
Instead of the sodium imino-diacetate there may be used, for example, an alkali salt of the N-methyl- or N-ethyliminodiacetic acid or of the N-(ortho-carboxyphenyl)imino-diacetic acid or of the C,C'-dimethyl-trimethylamine- α : α' : α'' -tricarboxylic acid and the like compounds. Moreover, instead of the alkali salts other water-soluble salts may be used, for example ammonium salts or salts of organic bases; also mixtures of several such substances may be used.

6. A desizing bath is made by dissolving 5 parts of malt amylase and .5 part of ethylenediaminetetracetic acid in 1000 parts of water at about 50° C. The pH-value is adjusted to about 5.

Where in the examples special instructions are not found, the enzyme preparations used may be of the usual commercial composition. Highly concentrated preparations are especially suitable.

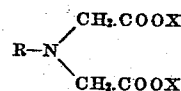
I claim:

1. A desizing agent which comprises amylase and a water-soluble compound of the formula:



wherein R₁ to R₅ stand for hydrogen, an aliphatic, cycloaliphatic or aromatic residue and X for a cation which does not disturb the action of the amylase.

2. A desizing agent which comprises amylase and a water-soluble compound of the formula:



wherein R stands for hydrogen, an aliphatic, cycloaliphatic or aromatic residue and X for a cation which does not disturb the action of the amylase.

3. A desizing agent comprising amylase and a water soluble alkali metal salt of trimethylamine- α : α' : α'' -tricarboxylic acid.

4. A desizing agent comprising amylase and a water soluble alkali metal salt of iminodiacetic acid.

5. A desizing agent comprising amylase and a water soluble alkali metal salt of ethylenediaminetetracetic acid.

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