Method for reducing the concentration of SO_3 in a reaction mixture comprising methane sulfonic acid and SO_3

The present invention refers to a method for reducing the concentration of SO_3 in a reaction mixture comprising methane sulfonic acid CH_3-SO_3H and SO_3. The method comprises a step of determining the amount of an additive needed to reduce the concentration of SO_3 by measuring density and/or conductivity of the mixture.

Alkane sulfonic acids are organic acids that can reach a similar acid strength as that of inorganic mineral acids, for example, sulfuric acid. However, in contrast to usual mineral acids such as sulfuric and nitric acids, the sulfonic acids are non-oxidizing and do not give off vapors that are harmful to health, as can be observed with hydrochloric and nitric acids. Further, many sulfonic acids, for example, methane sulfonic acid, are biologically degradable. The applications of sulfonic acids are many, for example, in cleaning agents, surfactants, as catalysts, and in organic synthesis, pharmaceutical chemistry, for example, as protective groups. The salts of sulfonic acids are employed, for example, as surfactants, for example, sodium dodecylsulfonate, or in the electroplating industry, especially as tin, zinc, silver, lead and indium, but also other metal alkylsulfonates. The very high solubility of alkyl sulfonates plays an important role, in particular. Further, no harmful gases are formed in electrolysis, and the use of toxic compounds, for example, cyanide, which is common in many cases, is dispensed with.

The structurally simplest representative of alkane sulfonic acids is methane sulfonic acid. Different methods for the synthesis are described in the prior art, for example in US 2,493,038, US 2005/0070614, WO 2007/136425 A2, EP 3071549 A1. In the prior art, methane and SO_3 are reacting with each other. The resulting reaction mixture comprises methane sulfonic acid as well as SO_3 as non-reacted educt. This SO_3 should be removed prior to distillation, as otherwise side products may occur (Robinson and Silberberg: "The reaction of methanesulfonic acid with sulfur trioxide", Canadian Journal of Chemistry, Vol. 44 (1966), 1437ff). Respective removal is for example disclosed in WO 2018/208701 A1 or European patent application 19176382.0. But even though there is the teaching that excess of SO_3 is to be removed, there is no teaching on how the amount of additive, needed to remove or at least significantly reduce the concentration of SO_3, is determined. Thus, there is the need for a method which enables the reduction of concentration of SO_3 in a liquid mixture in a fast and easy manner.

Surprisingly, it was found that conductivity and/or density of a mixture comprising methane sulfonic acid as well as SO_3 enables to determine the amount of an additive needed to significantly reduce the concentration of SO_3 in said mixture. The problem of the present disclosure is there

fore solved by a first aspect, in a method for reducing the SO_3 concentration in a mixture comprising methane sulfonic acid and SO_3, the method comprising:

i. providing a mixture comprising methane sulfonic acid and SO_3,
ii. determining the amount of an additive necessary to reduce the concentration of SO_3 in the mixture by forming a reaction product between the additive and SO_3,
iii. introducing said predetermined amount of the additive into the mixture;
iv. optionally repeating steps ii. and iii.,

wherein the determination of the amount of additive necessary to reduce the concentration of SO_3 in the mixture is performed by measuring conductivity and/or density of the reaction mixture, and wherein the additive preferably comprises water.

The mixture provided in step i. may be a reaction mixture resulting from a method of producing methane sulfonic acid from methane and SO_3 as described in the prior art. Preferably, it is a method as disclosed in EP 3071549 A1 and/or European patent application 19176382.0. In these cases, the mixture is a reaction mixture comprising methane sulfonic acid, methane and SO_3 as non-reacted educts, sulfuric acid as well as eventually occurring side products. In a preferred embodiment, providing of step i. comprises:
in a first step reacting methane sulfonic acid and hydrogen peroxide with each other to form a compound according to the following formula (I)

$$ALK\text{-}SO_2\text{-}O\text{-}O\text{-}X \qquad (I)$$

wherein ALK is methyl and X = hydrogen. This compound according to formula (I) is then provided in a reactor together with sulfur trioxide and methane. Due to the addition of the methane, a pressure within a range of from 1-200 bar is set. The reaction mixture is inside a high-pressure reactor and the temperature is controlled to be within a range of from 0° C to 100° C. Due to the reaction of the components to with each other, drop of the pressure can be monitored. This reaction mixture comprises the reaction product, namely the alkane sulfonic acid, as well as non-reacted educts, which are hydrocarbon and sulfur trioxide.

SO_3 may be provided as pure SO_3 or as oleum within the meaning of the present invention.

The additive is a compound which is able to react with SO_3 forming a compound preferably with a boiling point different from that of methane sulfonic acid. Preferably, the additive comprises water so that sulfuric acid is formed. The additive is a compound which is able to react with SO_3 forming a compound preferably with a boiling point different from that of the sulfonic acid. Preferably, the additive comprises water so that sulfuric acid is formed. If necessary, sulfuric acid or any other reaction product of SO_3 and the additive may be separated from the sulfuric acid in a