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Research Article

Synthesis of Synergist Antioxidant by Greener and Economical Methods

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ABSTRACT

Various types of antioxidants are useful in industries for stabilization of dispersions and other formulations. With the increase in demand for the synthesis of new antioxidants the methods for the synthesis of these antioxidants have also become equally important. The antioxidant Propanoic acid 3-(dodecylthio)-,oxybis (2,1-ethanediyoxy-2,1-ethanediyl) ester is the one which prevents the tanning and decomposition of the latex moreover it acts or enhances the antioxidant properties when combined with other phenolic antioxidant. The economical and cost-effective way of synthesis is mentioned in the paper.

Keywords: Antioxidant, NDM, methyl methacrylate.

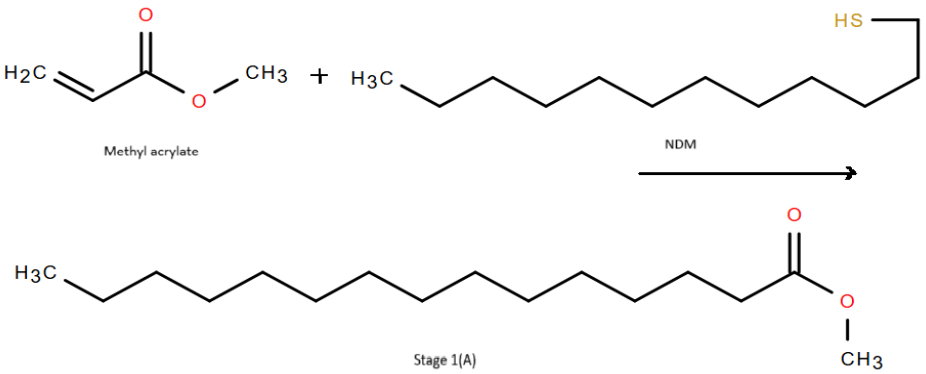
INTRODUCTION

Antioxidants are known to protect the composition of the various formulations thus preventing it from degradation and also increase the components shelf life.There are various types of the antioxidants that are available which are known to prevent the decomposition and decolorization of the components in the latex, particularly when used in the rubber manufacturing latex .Rubber products are well known to everyone and their uses differ from manufacturing of gloves to rubber tyres. So whatever be the usage the rubber products must be stable under given conditions. In Other words the components in the rubber must be stable under a given conditions so that performance is not affected. And the self life is improved. For this purpose antioxidants are used, which serves two purposes one it maintains the color of the latex and the other prevent it from the unwanted oxidation by products which is formed when latex is exposed to air or oxygen. Sometimes it is also found that the activity of the antioxidant is enhanced by addition of another component. This property is known as synerist. For the antioxidation of the latex various antioxidants are known. Some of these are phenolic while the other might be sulphur based. The main mechanism by which these antioxidants work is they absorb oxygen and form oxide or other form thus preventing the latex from decomposition. One of the known compounds to enhance the antioxidant properties of the antioxidant is Propanoic acid 3-(dodecylthio)-,oxybis(2,1-ethanediyoxy-2,1-ethanediyl) ester. The Compound very popularly known as wingstay SN are known to enhance the antioxidant properties of the phenolic antioxidants used in the latex.

With the known benefits of the antioxidants, the route of synthesis becomes equally important because the antioxidants needs to be cheap and also pure. The purity of the antioxidant is important because if the antioxidant is impure it will not be as effective antioxidant when compared to the pure antioxidant. So route designed to synthesize Propanoic acid 3-(dodecylthio)-,oxybis(2,1-ethanediyoxy-2,1-ethanediyl) ester needs to be free from the impurities and if formed must be easily removable. The route of synthesis that is mentioned in the paper is relatively simple and free from byproducts.

2. RESULTS AND DISCUSSION

The designed route of synthesis mentioned in the discussion involves two steps. First step is the addition reaction and the next reaction is the exchange reaction. In addition reaction ,the atom economy is high because all the starting material is being incorporated into the products. In the first step, there is reaction between n dodecyl mercaptan and methyl acrylate in the presence of a suitable quaternary ammonium based catalyst. The following scheme shows the reaction between the NDM and methyl acrylate.



When the reaction was carried out without the catalyst it was found that the reaction takes a longer time and also the reaction also doesn't go to completion. Study was done with and without catalyst and it was found that the reaction goes faster with the catalyst.

The following table shows the effect of the catalyst on the rate of the reaction:

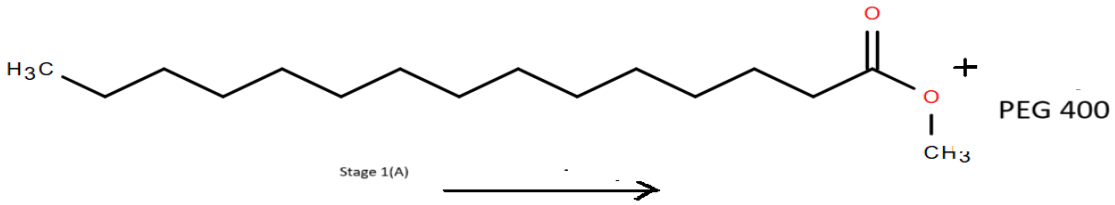
Sr no	NDM (mg)	Methyl methacrylate (mg)	Catalyst (mg)	Digestion Time (Hrs.)	Digestion Temp (°C)
1	1100	2500	Nil	9	100
2	1100	2500	0.1	9	100
3	1100	2500	10	9	100
4	1100	2500	100	9	100

The GC conversion of the reaction are as follows:

Sr no	Unreacted NDM (%)	Unreacted methyl acrylate (%)	Low Boiler (%)	Main Product	High Boiler (%)
1	35	45	10	10	nil
2	21	23.35	5	50	0.65
3	5	7.3	7	80	0.7
4	0.4	0.7	2	96	0.9

It is clear from the above table that with the use of the catalyst, which in this case is quaternary ammonium salt, the rate of reaction also increases, and the reaction goes to completion.

The second step of the reaction is the reaction between Stage I product (A) and the propylene glycol (PEG). The figure shows the reaction in detail.



Propanoic acid 3-(dodecylthio)-,oxybis(2,1-ethanediyoxy-2,1-ethanediyl) ester

Propylene glycol is generally known to come in various grades which range from PEG 200 to PEG 400.

In our study PEG 400 is used. Here the actual meaning of 400 means that the average molecular weight of glycol chains. When PEG 400 is reacted with the Stage I(A) product in the presence of the Tyzor TNBT (Dorf Kettle catalyst) at high temperature under atmospheric pressure the amount of methanol recovered is calculated. The amount of methanol distilled out is calculated from its theoretical value.

The following table gives the relation between the quantity of Tyzor TNBT vs the rate of methanol recovery (which is indicative of the rate of reaction).

Sr no.	Stage I (A) (mg)	Propylene Glycol (mg)	Tyzor TNBT (mg)	Digestion Time (Hrs.)	Digestion Temp (°C)	Methanol Recovered (mg)
1	600	200	nil	10	180	nil
2	600	200	0.015	10	180	22
3	600	200	0.02	10	180	35
4	600	200	0.03	10	180	42

From the table it is clear that only a small amount of Tyzor TNBT is required for the reaction to take place and there is no need for the separation of the catalyst.

3. INSTRUMENTAL SECTION:-

The instruments required for the analysis is

1) GC 2014 of Shimadzu.

2) HPLC (waters).

3) Distillation assembly and glassware set up.

4) GC MS of molecular weight identification.

5) GPC.

4. EXPERIMENTAL SECTION: -

Stage 1: -Known quantity of NDM and methyl acrylate was mixed together and Triton B, which is taken as a catalyst ,was added. The reaction was stirred at RT for 4 Hrs. and then was heated at 80 C. Initially the methyl acrylate starts refluxing and later on it stops which indicates the end point of the reaction. The completion of the reaction is indicated by the disappearance of the methyl acrylate peak.

Stage 2: -Two moles of stage 1 and one mole of PEG 400 was added in the flask equipped with the stirrer and 0.001% of tyzor TNBT was added as a catalyst and the reaction mixture was heated at 180 and simultaneously methanol was distilled out. About two mole of methanol was distilled out. The quantity of methanol indicates the end point of the reaction.

5. CONCLUSION

There are various ways to synthesize a molecule but nowadays the main focus is on synthesizing the molecule with high atom economy and less effluents. The process mentioned in the paper is clean, economical and also without any effluents thus greener.

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