Sulfamic acid/magnesium sulfate promoted esterification reactions under solvent-free and microwave-assisted conditions

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Introdução

The zwitterionic sulfamic acid [NH_2SO_3H, (SA)] is a strong inorganic acid, existing as a white crystalline solid, witch is non-hygroscopic, nonvolatile, odorless and inexpensive and readily commercially available. Because their properties, SA is a strong candidate to promote acid-catalyzed reactions, and has been used as heterogeneous catalyst in many organic transformation, including esterification reactions.[1]

Besides being classic and well-researched transformation, the esterification reactions are one of the most important organic transformations, leading to valuable chemicals, such as flavors, cosmetics, lubricants, pharmaceuticals, and plasticizers.[2]

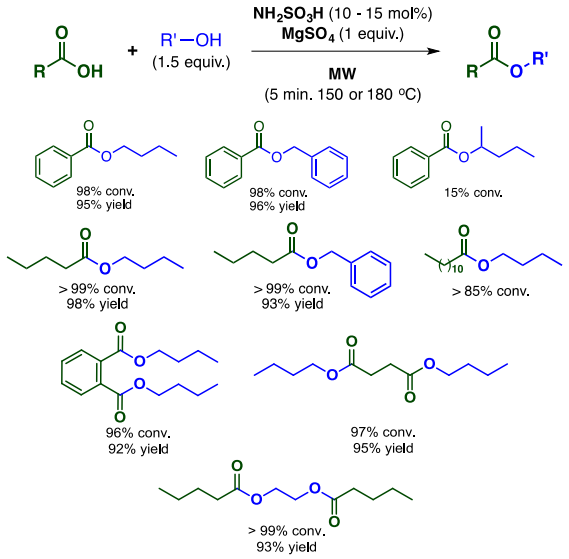
Successfully esterification reactions promoted by NH_2SO_3H were reported using acyl chlorides and anhydrides as acylating agents.[3] However, for carboxylic acids long reactions time is required to drive the reaction to completion. In this way, we explored the properties of microwave heating to enhance reaction rates.

Resultados e Discussão

In the present study, we investigated the best conditions to obtain the desired esters using a mono-mode laboratory instrument (Anton Paar, Monowave 300). We evaluated different temperatures, reaction times and catalyst loading to achieve the maximum conversion. Moreover, for the success of the esterification reaction, the water formed in the course of the reaction must be trapped. Among the assessed desiccant agents, $MgSO_4$ lead to the best results.

The esters from benzoic acid were obtained with up to 98% of conversion and good yields in 5 minutes of reaction at 180 ºC. Because the solid nature of the benzoic acid, 1.5 equivalents of the alcohol partner is necessary to achieve higher conversions. Secondary alcohols such as 2-pentanol lead to desired ester in only 15% of conversion.

The esterification reactions of the pentanoic acid with different alcohols were accomplished in 5 minutes at 150 ºC. Long-chain saturated fatty acids like lauric acid was also esterified under the same reaction conditions with conversion up to 85%.



Scheme 1. Carboxylic esters obtained by NH2SO3H/ MgSO4 system under microwave heating.

The present methodology is also valuable for the esterification of dicarboxylic acids like succinic or phthalic acid and double esterification reaction of diols such as etileneglycol.

Conclusões

In summary, we have developed a simply and efficient green method for esterification reactions Good to excellent yields were achieved in a short time We believe that this methodology will be a valuable addition to the existing methods in the field of esterification reactions.

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