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A NOVEL SYNTHESIS OF AMINO ACID ESTER USING MICROWAVE IRRADIATION

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ABSTRACT

A straightforward and innovative approach to the synthesis of a number of amino acid esters is reported here. This approach involves the presence of solid acid and microwave irradiation, as well as the combination of an amino acid and alcohol.

Keywords: Microwave Irradiation, Amino acid ester.

1. INTRODUCTION

Utilizing solid acids such montmorillonite clays [1,2], alumina [3,4] and silica gel [5], in conjunction with microwaves [6,7], is a method that is beneficial to the environment for the synthesis of organic compounds. The most significant benefit is related to the instantaneous localized superheating of materials in a manner that is both homogeneous and selective [8]. These results in a significant reduction in the amount of time required for reaction. Irradiation with microwaves has been used in peptide and protein chemistry, however there are just a handful of first studies on its application [9,10]. The use of microwave ovens in the solid phase synthesis of acyl carrier protein fragments 65–74 was also accomplished [10]. This was accomplished by employing Fmoc-amino acids employing either symmetrical anhydrides or active esters for coupling, which resulted in a significant reduction in reaction time (2-6 min) in comparison to traditional coupling procedures. Additionally, the yield was achieved using microwave ovens. This, using solid acids and microwave irradiation, this brief work discusses the efficient, easy, and fast synthesis of amino acid ester **3**.

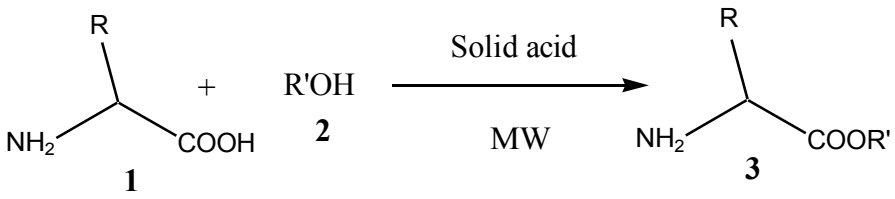
2. EXPERIMENTAL

The Leitz-Wetzlar melting point equipment was used to determine the melting points, and the results have not been corrected for any errors. The infrared spectra were captured by a Nicolet Impact 400D infrared spectrometer, which was equipped with KBr pellets and had a resolution of 4 cm−1. For the purpose of obtaining 1H NMR spectra, a Bruker AACF 400 MHz spectrometer was utilized and Me4Si was utilized as the internal standard. All of the studies were carried out with a commercial microwave oven (LG little chef model 194A) that was designed for residential use and operated at a frequency of 2450 MHz.

Synthesis of amino acid ester: In a 100 mL glass beaker, 3 mL alcohol, 10 mmol of amino acid, and 1 g of solid acid were combined and carefully mixed. A microwave irradiation operating at its 40% power level was applied to the beaker that contained the reaction mixture. In order to bring the reaction mixture back down to room temperature when the reaction had finished, it was chilled. An ether-based solvent was used to dissolve the product, and then celite was used to filter the mixture.

3. RESULTS & DISCUSSION

Through the utilization of solid acids like montmorillonite clays, alumina and silica gel in conjunction with microwave irradiation, the synthesis of amino acid ester (**Scheme-I**) has been carried out in a straightforward manner in this brief communication. After placing the mixture of amino acid, alcohol, and solid acid in a beaker, the esterification reaction was carried out by exposing the mixture to microwaves in a domestic microwave oven that had not been modified and was operating at a frequency of 2450 MHz with a power of forty percent. TLC analysis in a solvent system BAW (*n*-butanol, acetic acid, water:: 4:1:1) reveals that the ester production is finished in around 2-3 min. This is determined by the fact that the ester formation is monitored. The mixture that was produced as a result of the reaction was brought down to room temperature and then dissolved in ether used as a solvent in order to obtain the desired product with yields of approximately 70–75%. There were three distinct solid acids that were used to test the reaction, and each of them produced a different quantity of yield. After doing the experiment, it was discovered that the montmorillinite clay was the most appropriate reagent for the substrates that were being examined.



Scheme-I

The product formation was studied by the use of IR and NMR spectroscopy, and by comparing its melting point with the information available in the literature. The identification of a distinct peak corresponding to the stretching of the C=O bond in the infrared spectrum of the ester, occurring at approximately 1205–1705 cm^{-1}, provided confirmation of the occurrence of etherification. Their characterization was further validated by their ^{1}H NMR study. The ^{1}H NMR data obtained from all the compounds was excellent. Various R and R' groups have been used to synthesize many amino acid esters, following a similar process (Table-1).

Table 1

S.No.	R	R′	Yield*			Time (min)
			Clay	Alumina	Silica	
1.	H	CH_3	75	61	55	2
2.	H	C_2H_5	73	62	54	2
3.	H	Benzyl	71	63	57	2
4.	CH_3	CH_3	70	62	49	2
5.	CH_3	C_2H_5	74	61	51	2
6.	CH_3	Benzyl	75	64	49	2

*Isolated Yield

Conclusions

In short, the traditional procedure for producing esters of amino acids, which entails heating a mixture of amino acid, sulphuric acid and alcohol under reflux conditions for approximately 3-4 h, has been replaced. The current investigation has discovered that amino acid esters can be easily synthesized through the use of microwave irradiation. The technique is characterized by its speed, effectiveness, and simplicity. Furthermore, the returns are very commendable.

REFERENCES

1. P. Laszlo, *Science*, *235*, 1473, **1987**.
2. S.M.S. Chauhan, R. Singh, Geetanjali; *Synth. Commun*. *33*(7), 1179, **2003**.
3. G.H. Posner; *Angew Chem Int Engl 17*, 487, **1978**.
4. V.S. Joshi, N.P. Damodaran, S. Dev; *Tetrahedron*, *24*, 5817, **1968**.
5. J.H. Clark, J.C. Ross, D.J. Macquarrie, S.J. Barlow, T.W. Bastock, *Chem. Commun.*, 1203, **1997**.
6. R. Pazik, D. Hreniak, W. Strek, *Mater.s Res. Bull. 42*(7), 1188, **2007**.
7. R.A. Abramovitch, *Org. Prep. Proc. Int*., *23*, 683, **1991**.
8. S. Cresswell and S.J. Haswell, *Chem. Indus*., 621, **1999**.
9. S.-T. Chen, S.-H. Chiou, Y.-H. Chu and K.-T. Wang, *Int. J. Peptide Protein Res*., *30*, 572 **1987**.
10. H.M. Yu, S.-T. Chen and K.-T. Wang, *J. Org. Chem*., *57*, 4781, **1992**.