

Synthesis of β -enaminones catalyzed by nanoparticles of Fe₂O₃ in ultrasound and solvent-free approach.

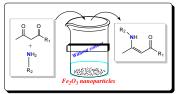
Cristiane Regina Winck Hortelan (PG)^{1,2}, Ingridhy Ostaciana Maia Freitas da Silveira (PG)², Silvia Mendes de Souza (IC)¹, Adilson Beatriz (PQ)², Roberto da Silva Gomes (PQ)², Nelson Luís de Campos Domingues (PQ)¹.

¹Federal University of Grande Dourados-Hybrid Materials Laboratory- HML ²Federal University of Mato Grosso do Sul, Institute of Chemistry *cris_winck@hotmail.com

Keywords: β-enaminonas, nanopartículas, ultrasound.

INTRODUCTION

Nowadays, there's been growing the interest of some researches groups by nanosized metal particles due to their high catalytic activity compared to their bulk counterparts¹. The result from the heterogenic catalysis using nanosized metal particles is environmentally desirable². Furthermore, the solvent absence as well as the use of ultrasound make this methodology in accordance with the Green Chemistry protocols. Among the compounds nowadays synthesized, β -enaminones are the ones that present a great synthetic interest as building blocks or bioactives³. This way, the aim of this study is the synthesis of β -enaminones catalyzed by nanoparticles of Fe₂O₃ through a clean methodology (Scheme 1).



Scheme 1. General procedure for β -enaminones synthesis.

RESULTS AND DISCUSSION

 Fe_2O_3 nanoparticle was synthesized as described by Pechini⁴ method and characterized by electronic transmission electron microscopy. Figure 1 shows a nanosized scale about 100 ± 10 nm. It is also possible to observe the crystallinity of the synthesized material indicating the success of the synthesis methodology.

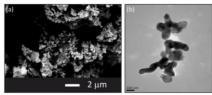


Figure 1. MET micrographs of Fe_2O_3 synthetized nanoparticles.

The nanosizes Fe_2O_3 was inserted on β -enaminones synthesis and the data are presented in table1.

Table 1. β -enaminone synthesis in the presence of Fe₂O₃ nanoparticles under ultrasonic irradiation.

Entry	Dicarbonyl	Amine	Yield*
,	compound		(%)
	compound		(70)
1	Ethyl acetoacetate	<i>p</i> -anisidine	87
	Elligi aceluacelale	<i>p</i> -anisiune	07
2	Ethyl acetoacetate	Benzylamine	84
	-	2	
3	Ethyl acetoacetate	Butylamine	80
5	Lifyraceloacelale	Datylamine	00
4	Ethyd a sata a satata	Cueleboxulamina	00
4	Ethyl acetoacetate	Cyclohexylamine	83
-			
5	Ethyl acetoacetate	<i>p</i> -toluidine	91
	-	-	
6	Ethyl acetoacetate	aniline	56
Ũ		armite	00
7	agetylegetene	<i>p</i> -anisidine	86
/	acetylacetone	<i>p</i> -anisiume	00
8	acetylacetone	Benzylamine	60
9	acetylacetone	Butylamine	35
	,	,	
10	acetylacetone	Cyclohexylamine	64
.0	accignatione	-,	~
- 44		6.1.2.12	
11	acetylacetone	<i>p</i> -toluidine	92
*All products were characterized by RMN of ¹ H and ¹³ C.			

All products were characterized by RMN of 'H and 'SC.

CONCLUSION

The methodology used in the synthesis of β enaminones proved to be very efficient according to the high yields obtained, low reaction time and mild reaction conditions.



Larisa B. Arruda, Marcelo O. Orlandi, , Paulo Noronha Lisboa-Filho, Ultrasonics Sonochemistry 2013, 20, 799–804. David Ian MaGee,a,*Minoo Dabiri,b* Peyman Salehi,c* and Laleh Torkian ARKIVOC 2011, xi, 156-164.

A. Venkat Narsaiah*, A.R. Reddy, B.V.S. Reddy and J.S. Yadav. Amberlyst-15: An Efficient, Cost-Effective and Recyclable Heterogeneous Solid Acid Catalyst for the Synthesis of β -Enaminones and β -Enamino Esters. The Open Catalysis Journal, 2011, 4, 43-46.

Pechini, N., U.S. Patent, 1967, 3, 330-697.