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Synthesis of 3,4-dihydrobenzo[2,3-*d]* pyrimidines using K-10 clay as catalyst under microwave ir-radiations (Part-1)

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ABSTRACT

The 8-cyano-4,5,6,7-tetraphenyl-3,4-dihydrobenzo[2,3-d]pyrimidine is synthesized by the condensation of 2-amino-1-benzoyl-3-cyano-5-hydroxy-4,5,6-triphenyl-1,3-cyclohexadiene and formamide using K-10 clay as catalyst under microwave ir-radiations.

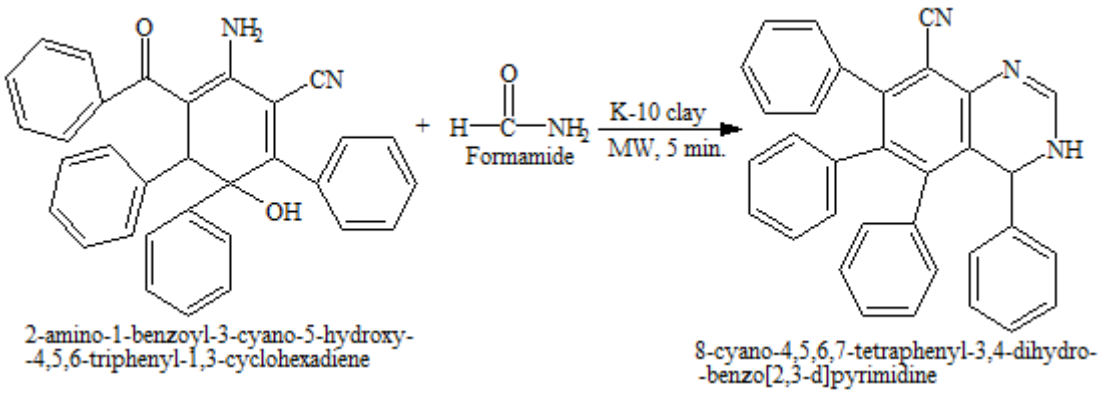
Keywords: Pyrimidines, K-10 clay, microwave, formamide.

INTRODUCTION

The heterocyclic compound i.e. benzopyrimidines have found application in a wide range of medicinal chemistry because of their diverse biological activities, such as antibacterial,[1,2] anticonvulsant, [3] antiinflammatory,[4–6] antitumor[7–9] and antifungal[10] activities. These chemotherapeutic applications of benzopyrimidine derivatives prompted us to synthesize some new highly substituted 3,4-dihydrobenzo[2,3-*d]*pyrimidines by adopting a new route under solid supported microwave irradiations.

Several methods have been reported[11–14] for the synthesis of benzopyrimidine derivatives. However, these methods suffer from drawbacks, such as longer reaction time, complicated workup, use of expensive and hazardous chemicals with low yield. The title compounds were synthesized in this study using commonly available reagents under dry media microwave irradiation to overcome the mentioned drawbacks. Microwave assisted organic synthesis proceeds with facile reactions to provide high yield within a very short reaction time period.[15] This methodology also avoids the use of excess solvents and harmful acids or bases, which are generally used for the catalysis of the reactions.[16–19] Solution phase microwave organic reactions have some limitations, such as the possibility of super heating of the solvents which may result in series explosions.[20,21] Microwave activated dry media synthesis on solid inorganic supports is the most efficient and ecofriendly technology.[22,23] Reactions can be carried out easily at ambient pressure in open vessels by using domestic microwave ovens.[24] Moreover, the use of solid acid and base catalysts reduces the amount of toxic wastes and by-product formation.[25]

The pharmaceutical interest in benzopyrimidines prompted us to synthesize the 8-cyano-4,5,6,7-tetraphenyl-3,4-dihydrobenzo[2,3-d]pyrimidine using K-10 clay as catalyst under microwave ir-radiations. A mixture of 2-amino-1-benzoyl-3-cyano-5-hydroxy-4,5,6-triphenyl-1,3-cyclohexadiene and formamide condensed using K-10 as catalyst under microwave irradiation in a few minutes. (scheme-1). The crude product is crystallized from methanol to give about 75% yields.



Scheme-1

The product was identified on the basis of their IR, ^{1}H-NMR, and by comparison of their Rf values with those of authentic samples prepared by standard routes.

Conclusion

In conclusion, we have developed a new and efficient method for the formation of 8-cyano-4,5,6,7- tetraphenyl-3,4-dihydrobenzo[2,3-d]pyrimidine in high yields in a short time using K-10 as catalyst under microwave irradiation.

Typical experimental procedure

The 2-amino-1-benzoyl-3-cyano-5-hydroxy-4,5,6-triphenyl-1,3-cyclohexadiene (0.01 mol), formamide(10 ml), K-10 clay(1g) taken in the open erlenmyer flask (100ml). The flask was subjected to 70% irradiation level (560W) in the Kenstar OM-9925 (800W) unmodified domestic microwave oven operating at 2450 MHz for 5 minutes. The progress of the reaction was monitored every 30 s. The product was extracted with ethanol (4 x 10 ml) and obtained in the solid state after removal of solvent by distillation under reduced pressure. The product was recrystallized from MeOH. **Melting point:** 157°C; **^{1}H-NMR**(δ in ppm in $CDCl_3$): 4.6 (*s*, 1H, C-4), 7.1–7.2 (*m*, 2oH, Ar–H & 1H at C-2), 11.5 (*brs*, 1H, NH);and **IR** (KBr, cm^{-1}) 1600 (C=N), 3350 (N–H), 2240 (Ca≡N)cm^{-1}.

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