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

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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 KSF clay as catalyst under microwave ir-radiations.

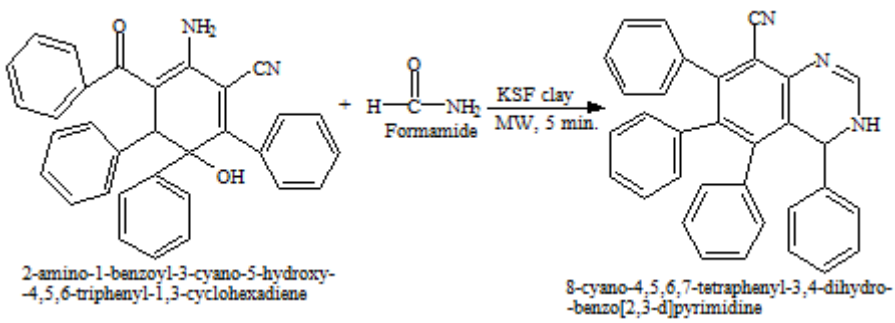
Key words: Pyrimidines, KSF clay, microwave, formamide.

INTRODUCTION

The pyrimidines has wide range of medicinal chemistry such as antibacterial,[1,2] anticonvulsant, [3] antiinflammatory,[4–6] antitumor[7–9] and antifungal[10] activities. These 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 substituted pyrimidine derivatives. However, these methods suffer from drawbacks, such as longer reaction time, complicated workup, use of expensive and hazardous chemicals with low yield. The 3,4-dihydrobenzo[2,3-*d]*pyrimidines was synthesized in this study using commonly available reagents under dry media microwave irradiation to overcome the mentioned drawbacks.[15–25]

In previous paper[26], we synthesized 8-cyano-4,5,6,7-tetraphenyl-3,4-dihydrobenzo[2,3-d]pyrimidine using K-10 clay as catalyst under microwave ir-radiations. Now we want to explore our work for the synthesis of 8-cyano-4,5,6,7-tetraphenyl-3,4-dihydrobenzo[2,3-d]pyrimidine using KSF 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 KSF as catalyst under microwave irradiation in a few minutes. (scheme-1). The crude product is crystallized from methanol to give about 76% yields. The product was identified on the basis of their IR, ^{1}H-NMR, and by



Scheme -1

comparison of their Rf values with those of authentic samples prepared by standard routes.

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), KSF 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*, 20H, 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}.

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 KSF as catalyst under microwave irradiation.

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