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Study the effect of change in microwave power level in organic reactions using heterogenous Mont. K-10 catalyst

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ABSTRACT

The effect of change in power level of microwave ir-irdation on product yield in organic reaction using heterogeneous Mont. K-10 catalyst in explored.

Key words: Mont. K-10, clay, catalyst, microwave, power level.

INTRODUCTION

Researches in organic chemistry are being explored throughout the world[1-6]. There are numerous reports about new reactions are performed, new techniques, new compounds etc., with the new techniques; many reactions are performed with high reaction rate, high selectivity and high percentage yield. Examples of these new techniques are the use of ultrasonic radition[7-9], catalyst[10-11] and microwave ir-radiation[12-13]. There is several publications report about the reactions under microwave ir-radiation which give high reaction rate and percentage yield. Therefore our research unit is established in order to study and develop the application of domestic microwave oven in organic chemistry reactions. The first paper on the use of microwave for the synthesis reactions appeared in the open, peer reviewed literature in 1986[12-14]. We already have been published our work as can seen in literature[18-23].

We now report the effect of change in microwave level on the product yield during the synthesis of acetals and terraphenylporphyrin using environmentally benign and mild acidic heterogeneous Mont. K-10 catalyst in dry acid media. The heterogonous Mont. K-10 catalyst has gained importance due to simple setups, work ups along with high purity, lower chemical degradation and high product yield.

The work was initiated by investigating the reaction in between benzaldehyde and pyrrole in the presence of Mont. K-10 clay catalyst in an open Erlenmeyer flask at 80W level of microwave ir-radiation in an unmodified microwave oven and the tetraphenylporphyrin (TPP) obtained in very-2 low yield. The same reaction is then carried out 160W, 240W, 320W, 400W, 480W, 560W, 640W, 720W and 800W and the best yield of around 30% was obtained only at 560W after 5 minutes (TLC monitoring) as shown in Table-1. Similarly, the same project is proved by carried out the reaction in between 4-methoxybenzaldehyde and ethylene glycol in the presence of Mont. K-10 catalyst using microwave ir-radiation level (i.e. 160W, 240W, 320W, 400W, 480W, 560W, 640W, 720W and 800W) and the best yield of around 89% was obtained only at 560W after 12 minutes. The result is given in Table-2.

Table -1: Synthesis of Tetrahydroporphyrin using Mont. K-10 catalyst under various ir-radiation (power levels)

S. No.	Power levels (In watts)	Yield (%)	Time (min)
1.	80	2	10
2.	160	6	10
3.	240	11	9
4.	320	16	8
5.	400	21	7
6.	480	28	5
7.	560	30	5
8.	640	26	5
9.	720	21	5
10.	800	15	5

Table -2: Synthesis of 2-(4-methoxy phenyl)-1,3-dioxalone using Mont. K-10 catalyst under various ir-radiation (power levels)

S. No.	Power levels (In watts)	Yield (%)	Time (min)
1.	80	38	17
2.	160	50	15
3.	240	57	15
4.	320	63	14
5.	400	71	14
6.	480	80	12
7.	560	89	12
8.	640	79	12
9.	720	72	12
10.	800	68	12

EXPERIMENTAL

Synthesis of Tetraphydroporphyrin (TPP)

In a typical procedure, Benzaldehyde (1m mole), Pyrrole (5m mole) and Mont. K-10 clay catalyst (1g) was taken in an Erlenmeyer flask (100 ml) and ir-radiate at 70% power level in an unmodified domestic microwave oven operating at 2450 MHz. After cooling to room temperature, the crude product extracted and recrystallized with ethanol to yield pure TPP. The product was identified on the base of their IR, ^{1}H-NMR and by comparison of their Rf values with those of authentic samples prepared by standard routes.

Synthesis of 2-(4-methoxy phenyl)-1,3-dioxalone

In a typical procedure, 4-methoxy benzaledhyde (5m mole), ethylene glycol (50m mole), Mont. K-10 clay catalyst (1g) was taken in an Erlenmeyer flask (100ml) and ir-radiated at 70% power level in an modified domestic microwave oven operating at 2450 MHz. The contents were transferred into water (50ml). The organic product was extracted into benzene (50ml) and dried over Na2SO4 (anhyd). The solvent was distilled off, giving the desired pure 2-(4-methoxy phenyl)-1,3-dioxalone. 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

We have shown that the product can be formed rapidly in high yield using Mont. K-10 catalyst at 560W microwave ir-radiation level in an unmodified domestic microwave.

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