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RESEARCH PAPER

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Effect of Changing in the Microwave power levels on Synthesis of Tetraphenylporphyrin by using different Heterogeneous Catalysts

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ABSTRACT

The synthesis of tetraphenylporphyrin (TPP) by the reaction between benzaldehyde and pyrrole in the presence of different heterogeneous catalysts Mont. KSF clay, Yb $(OTf)_3$, $Sc(OTf)_3$, $Dy(OTf)_3$ and $InCl_3$ at 80 W power level of microwave irradiation in an unmodified microwave oven was carried out at 160 W, 240 W, 320 W, 400 W, 480 W, 560 W, 640 W, 720 W and 800 W. The optimum yield was obtained only at 560 W power level (by TLC monitoring).

Keywords: ***heterogeneous catalyst, microwave, power level, pyrrole, tetraphenylporphyrin, TLC.***

INTRODUCTION

In the world researches in organic chemistry are being explored (Vogel, 1965, Corey and Mock, 1962, Hung and Rawl, 2000, Deyle and Phillips, 2001, Gainelli et.al. 2003 and Kobayashi et.al. 2003). There are numerous reports about new reactions 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 radiation (Horton et al. 1984, Yamashita and Inonnetal, 1985, Bar, 1988 and Ahluwalia et al. 1989) catalyst (Ahluwalia and Khandori, 1989, Gigues et al. 1986 and Caddick, 1985) and microwave ir-radiation (Gedye et al. 1986 and Abrambovith, 1991). There are several publication reports 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 open, peer reviewed literature in 1986 (Gedte et al. 1996 and Loupy, 1999).

We now report the effect of change in microwave level on the product yield during the synthesis of TPP using environmentally benign and mild acidic heterogeneous catalyst Mont. KSF clay, $Yb(OTf)_3$, $Sc(OTf)_3$, $Dy(OTf)_3$ and $InCl_3$ in dry acid media (Singh et al. 2007). The heterogeneous catalyst has gained importance due to simple setups, workups along with high purity, lower chemical degradation and high product yield (Singh et al. 2007).

MATERIAL AND METHODS

Synthesis of tetraphenylporphyrin (TPP)

In an Erlenmeyer flask (100ml), benzaldehyde (1 mmole), pyrrole and different heterogeneous catalyst (1g) was taken 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 extacted and recrystallized with ethanol to yield pure TPP.

Colour: Violet

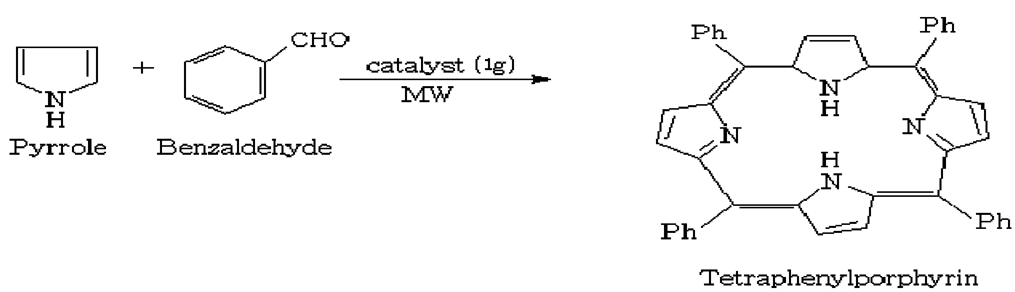
Melting point: 371 ^{O}C

^1H-NMR (δ in ppm in $CDCl_3$): 7.78 (12 H, *o* and *p* of phenyl), 8.24 (8H, pyrrole), 8.86 (8H, *m* of phenyl) and

IR (KBr) 3430, 3315, 3050, 3030, 1590, 1470, 1440, 780 and 690 cm^{-1}.

RESULTS AND DISCUSSION

The reaction in between benzaldehyde and pyrrole in the presence of Mont. KSF clay, $Yb(OTf)_3$, $Sc(OTf)_3$, $Dy(OTf)_3$ and $InCl_3$ catalyst in an open Erlenmeyer flask at 80W level of microwave ir-radiation in an unmodified microwave oven as shown in **Scheme-1**. The yield obtained is very low. The same reaction is then carried out 160W, 240W, 320W, 400W, 480W, 560W, 640W, 720W and 800W and the optimum yield of 35, 30, 32, 40 and 35 % was obtained only at 560W (TLC monitoring) as shown in **Table 1-5**



Scheme-1

Table 1. Synthesis of TPP using Mont. KSF catalyst under various ir-radiations (power levels).

S.No.	Power levels (In watts)	Yield (%)	Time (min)
1.	80	12	10
2.	160	16	10
3.	240	17	9
4.	320	16	8
5.	400	21	7
6.	480	28	5
7.	560	35	5
8.	640	26	5
9.	720	20	5
10.	800	18	5

Table 2. Synthesis of TPP using $Yb(OTf)_3$ catalyst under various ir-radiations (power levels).

S.No.	Power levels (In watts)	Yield (%)	Time (min)
1.	80	10	10
2.	160	16	10
3.	240	16	9
4.	320	18	8
5.	400	21	7
6.	480	28	5
7.	560	30	4
8.	640	26	6
9.	720	20	5
10.	800	18	6

Table 3. Synthesis of TPP using $Sc(OTf)_3$ catalyst under various ir-radiations (power levels).

S.No.	Power levels (In watts)	Yield (%)	Time (min)
1.	80	12	10
2.	160	10	10
3.	240	17	9
4.	320	16	8
5.	400	21	7
6.	480	26	5
7.	560	32	5
8.	640	26	5
9.	720	22	7
10.	800	18	7

Table 4. Synthesis of TPP using $Dy(OTf)_3$ catalyst under various ir-radiations (power levels).

S.No.	Power levels (In watts)	Yield (%)	Time (min)
1.	80	8	10
2.	160	10	10
3.	240	14	9
4.	320	16	8
5.	400	21	8
6.	480	28	7
7.	560	40	7
8.	640	26	7
9.	720	20	7
10.	800	20	7

Table 5. Synthesis of TPP using $InCl_3$ catalyst under various ir-radiations (power levels).

S.No.	Power levels (In watts)	Yield (%)	Time (min)
1.	80	15	10
2.	160	16	10
3.	240	17	9
4.	320	16	8
5.	400	20	7
6.	480	28	5
7.	560	35	5
8.	640	26	5
9.	720	20	5
10.	800	18	5

CONCLUSION

We have shown that the product can be formed rapidly in high yield using mild acidic heterogeneous catalyst Mont. KSF clay, $Yb(OTf)_3$, $Sc(OTf)_3$, $Dy(OTf)_3$ and $InCl_3$ at 560W microwave ir-radiation level in an unmodified domestic microwave.

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REFERENCES

Vogel, E, Grimme W. and Korte S. (1965) *Tetrahedron Lett.*, 21, 3625.
Corey, E.J. and Mock, W.L. (1962) *J. Am. Chem*. Soc., 84, 685.
Hung, Y. and Rawal, V.H. (2000) *Org. Lett.,* 2, 3321.
Deyle, M.P. and Phillips, I.M. (2001) *J. Am. Chem*. Soc., 123 5366.
Gainelli, G. Gallettti, P. Giacomini, D. and Quintavalla, A. (2003) *Tetrahedron Lett.*, 44, 93.
Kobayashi, S. Tanaka, H. Ami, H. and Uneyama, K. (2003) *Tetrahedron*, 59, 1547.
Horton, A.M. Hollinshead, D.M. and Ley, S.V. (1984) 40, 1737.
Yamashita, T. and Inoneetal, V. *Bull.Chem. Soc.*, 8, 2709.
Bar, R. (1988) *Biotechnol.*, 32, 655.
Ahluwalia, V.K. Adhikari, R. and Singh, R.P. (1989) *Synth. Commun.,* 15, 599.
Ahluwalia, V.K. and Khanduri, C.H. (1989) *Indian J.Chem.*, 28B, 599.
Gigues, R.J. Bray, T.L. Duncan M. and Majetich, G. (1986) *Tetrahedron Lett*., 27, 4945.
Caddick, S. (1995) *Tetrahedron*, 51, 10403.

Gedye, R. Smith, F. Westaway, K. Ali, H. Laberge, L. and Roussel, J. (1986) *Tetrahedron Lett.*, 27, 279.

Abrambovith, R. (1991) *Org. Prep. Proced. Int.*, 23, 683.

Gedye, R. Rank, W. Westaway, K.C. (1991) *Can. J. Chem.*, 69, 706.

Loupy, A. (1999) *Topic in current chemistry*, 206, 155.

Singh, R. Rani, M. and Malik, S. (2007) *Mat. Sci. Res. Ind.*, 4, 201.

Singh, R. Rani, M. Rani, S. and Malik, S. (2007) *Mat. Sci. Res. Ind.*, 4, 171.

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