Green Synthesis of Some Benzylidenes

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***Abstract*— The present paper present green synthesis of some benzylidenes and characterized by IR and NMR spectroscopy. The spectral data confirms the formation of benzylidenes. Earlier studies reported the synthesis of some benzylidenes by conventional methods. The green method of synthesis was found more effective in the sense of yield and reaction time as it reduces the reaction time as well as increased the yield of desired compounds.**

1. INTRODUCTION

Green chemistry interested for alternative innovative aspects of synthesizing organic compounds. It can be brought about by changing the techniques or less hazardous chemicals for synthesizing desired compounds [1-2]. Green chemistry leads to minimize the waste materials as well as formation of biodegradable by-products. In earlier days, conventional source of heat energy were used for transfer energy for carrying chemical reactions. In the course of development, number of heat energy sources were developed which effectively reduces the reaction time, increases yield of the product, minimizes the production of byproduct etc. In view of it this article reports green synthesis of some benzylidenes.

Synthesis of new chemical entities is major bottleneck in drug discovery. Conventional methods for various chemical syntheses is very well documented and practiced. The methods for synthesis (Heating process) of organic compounds has continuously modified from the decade. In 1855, Robert Bunsen invented the burner which acts as energy source for heating a reaction vessel, this was later superseded by isomental, oil bath or hot plate, but the drawback of heating, though method remain the same. Microwave Assisted Organic Synthesis (MAOS), which has developed in recent years, has been considered superior to traditional heating.

Microwave assisted organic synthesis (MAOS) has emerged as a new “lead” in organic synthesis. The technique offers simple, clean, fast, efficient, and economic for the synthesis of a large number of organic molecules. In the recent year microwave assisted organic reaction has emerged as new tool in organic synthesis. Important advantage of this technology include highly accelerated rate of the reaction, Reduction in reaction time with an improvement in the yield and quality of the product.

This synthetic technique has been based on the empirical observation that some organic reactions proceed much faster and with higher yields under microwave irradiation compared to conventional heating. In many cases reactions that normally require many hours at reflux temperature under classical conditions can be completed within several minutes or even seconds in a microwave oven, even at comparable reaction temperatures.

It has long been known that molecules undergo excitation with electromagnetic radiation. This effect is utilized in household microwave ovens to heat up food. However, chemists have only been using microwaves as a reaction methodology for a few years. Some of the first examples gave amazing results, which led to a flood of interest in microwave-accelerated synthesis.

The water molecule is the target for microwave ovens in the home; like any other molecule with a dipole, it absorbs microwave radiation. Microwave radiation is converted into heat with high efficiency, so that "superheating" becomes possible at ambient pressure. Enormous accelerations in reaction time can be achieved, if superheating is performed in closed vessels under high pressure; a reaction that

takes several hours under conventional conditions can be completed over the course of minutes.

Benzylidenes are important class of heterocyclic compounds due to their wide range of bioactivities and their applications in the field of drug research [3-16]. The schematic representation of its sysnthesis is shown in Fig. 1. They are used as antituberculotic agent [3], antibacterial agents [4], and antifungal agent [5].

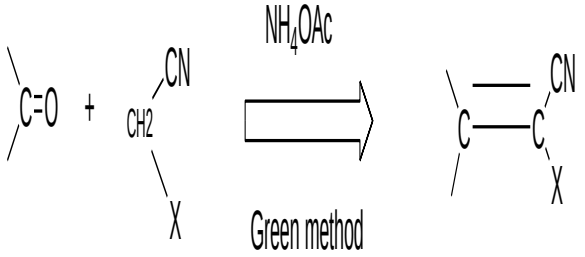

Fig. 1 Schematic representation of synthesis of Benzylidenes

Table 1: Synthesis of some benzylidenes catalyzed by ammonium acetate by green method

Compound	Abbreviation	M.P.	IR	^{1}H NMR
2-Benzylidenemalononitrile	BMN-A	77^{o}C	m 3030, 2233, 1598;	d 7.75 (s, 1H) 7.35–7.68 (m, 5H)
2-(4-Dimethylamino) benzylidene) malononitrile	BMN-B	138 ^{o}C	m 2922, 2210, 1560	d 7.75 (s, 1H) 7.70 (d, 2H J = 7.3 Hz) 6.67 (d, 2H J = 7.6 Hz), 3.1 (s, 6H)
2-(4-Hydroxy-3-methoxybenzylidene) malononitrile	BMN-C	182 ^{o}C	m 3445, 2215, 1270	d 7.87 (s, 1H) 6.99–7.02 (d, 2H, J = 7.4 Hz), 7.20 (s, 1H), 3.86(s, 3H), 4.52 (s, 1H)
2-(4-Nitrobenzylidene) malononitrile	BMN-D	156 ^{o}C	m 3032, 2225, 1570	8.20 (d, 2H J = 7.5 Hz), 8.00 (d, 2H J = 7.2 Hz), d 7.91 (s, 1H)

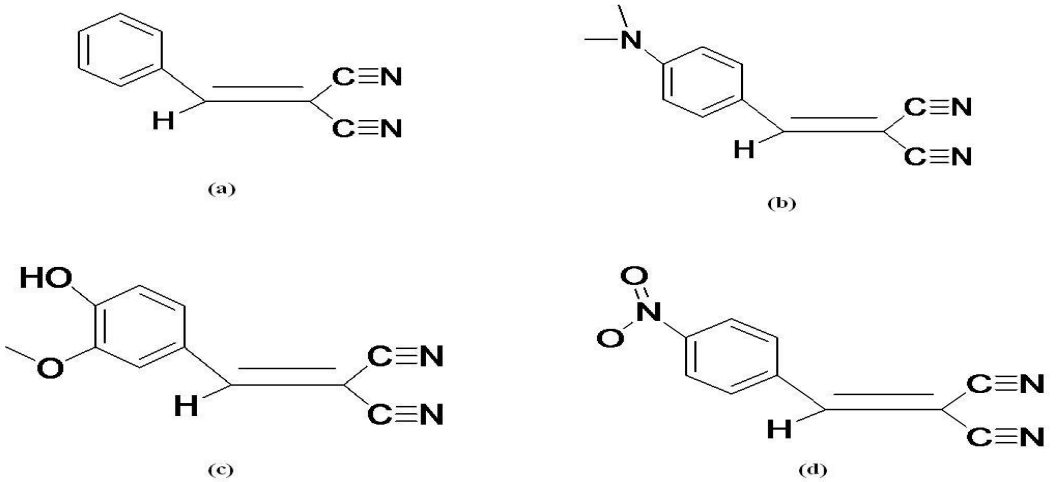

Fig. 2 Structure of synthesized compounds (a) 2-Benzylidenemalononitrile, (b) 2-(4-Dimethylamino) benzylidene) malononitrile, (c) 2-(4-Hydroxy-3- methoxybenzylidene) Malononitrile and (d) 2-(4-Nitrobenzylidene) malononitrile

2. EXPERIMENTAL SECTION

All melting points are measured in an open capillary and are uncorrected. IR spectra were recorded with a Shimadzu IR-408 spectrometer mr). 'H-NMR spectra were determined in DMSO solution on a FT-NMR Bruker AC-80 (80MHz). All the studied compounds were synthesized under the conditions as presented in Table 1. The structures of synthesized compounds are given as Fig. 1a-d.

3. RESULT AND DISCUSSION

It was found that these compounds were synthesized by conventional methods by refluxing with appropriate methods for 15 h. The application of microwave irradiation significantly reduces the reaction time up to 4-5 min and greatly enhanced the yield of product also. Thus, application of MW provides an alternative route for synthesizing these benzylidenes in maximum yield and in minimum time.

4. CONCLUSIONS

While exploring a green method of synthesis for benzylidenes as compared to conventional methods, it was found that MW assisted method was energy efficient as well as economical compared to conventional method of synthesis.

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