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EXPEDITIOUS GREEN SYNTHESIS OF VERSATILE ORGANIC COMPOUNDS BY DIVERSE METHODS

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1. Introduction:

Synthesis of organic compounds requires organic solvents and reagents to promote them. Very often, toxic solvents are used for this purpose. Many organic reactions require molar proportion of reactants and reagents to accomplish the goal. Some reactions require longer reaction times and produce products without control of stereochemistry. Therefore, development of facile and effective organic reactions under environmentally friendly conditions is necessary. In this perspective, several reactions are discussed that are performed by microwave-induced reactions, catalytic procedures, in the absence of any solvents, one-pot method and in water. Important reactions are chosen to investigate the feasibility of conducting these reactions under green chemistry conditions.

2. Results:

Microwave-Induced reactions:

The invention of microwave-mediated organic processes in 1986 has opened up significant challenge to diverse scientists and engineers. Microwave oven generates non-ionizing radiation that is responsible for dielectric heating of the reactants [1, 2]. This forces solvents and reactants to cause molecular interactions. At least one component of the reaction must be polar in this process. We have been pursuing microwave-mediated organic reaction for the past 27 years.

Kitchen and programmed microwave oven was used in our endeavor. Many molecules were prepared following microwave-induced reactions [3].

Synthesis of diverse beta lactams was performed by reaction with acid chloride (equivalent) and Schiff base in the presence of N-methylmorpholine or triethylamine. The beta lactams produced had *cis* or *trans* configuration. Diastereoselective and enantioselective reactions were also possible with certain specific substrates. Notably, the yields and stereochemistry of the beta lactams were not the same when microwave-mediated reactions and classical reactions were conducted in some examples. A high power microwave radiation caused to form the more stable product.

Microwave-induced hydrogenolysis of the benzyloxy group in beta lactams to the hydroxy beta lactams group was achieved with ammonium formate and 10% Pd/C. This reaction produced open chain amides due to the cleavage of N-C-4 bond. However, an aromatic ring directly connected to the C-4 position of the beta lactam is necessary for this cleavage reaction to occur. No cleavage of the N-C-4 bond was observed in the absence of an aromatic ring.

Microwave-induced methods have advantages over common heating systems when products are formed through acceleration of reaction following mild conditions, good selectivity and environmentally benign processes. There are some controversies regarding the rate acceleration in the microwave oven. Some authors believe it is the because of the rapid rise of temperature a reaction rate is enhanced. Some authors believe non-thermal effects are responsible for the acceleration of the reaction rate in most of the examples.

Stereoselectivity of beta lactam formation:

Stereochemistry plays the central role in controlling the medicinal activities of organic compounds. Development of stereospecific reactions, therefore, is the major challenge. Beta lactams are powerful medicines and medicinal activities depend on relative and absolute stereochemistry. Stereochemistry control in beta lactam synthesis is a part of green chemistry process. The formation of a single stereoisomer eliminates purification by chromatography and thus excessive solvents requirement for separation. It was found that *cis* hydroxy beta lactam derivatives are obtained at 0-5^{0}C by cycloaddition of acid chlorides and imines in the presence of a tertiary base (triethylamine). The same reaction at high temperature produced *trans* hydroxy beta lactam derivatives in major proportion (over 90%). In addition, by changing the conditions of the experiments, it was also possible to control stereospecificity of beta lactam synthesis. For example, a slow addition of a tertiary base (triethylamine or N-methylmorpholine) to a refluxing solution of a diarylimine with acid chlorides in toluene produced *trans* beta lactam. On the other hand, imines derived from carbohydrates and primary amines produced optically active *cis* beta lactams exclusively regardless of the choice of solvent. The stereochemical results remained identical when microwave irradiation was used for the preparation of these beta lactam derivatives. It was seen that low power microwave irradiation produces *cis* beta lactam in higher proportions than *trans* beta lactams. However, high power microwave-induced reactions of the same substrates produced *trans* beta lactams as major proportion. Pure *cis* beta lactams on exposure to high power microwave irradiation in the presence of tertiary base did not isomerizes

to *trans* beta lactams indicating that that the pathways for the formation of thermodynamically more stable *trans* beta lactams is different than *cis* beta lactam formation [3a, d, g, j, k].

Synthesis of Heterocycles by Microwave-induced Reactions:

Microwave-induced iodine-catalyzed reaction was performed for the synthesis quinazolines via condensation of 1, 2-dicarbonyl compounds and 1, 2-diamines [4a]. Tetrahydroquinolines were prepared *via* microwave induced reactions [4b]. Molecular rearrangement of beta-lactams to pyrrolidine was performed in methanol using microwave-induced reaction [4c]. Microwave-Assisted preparation of pyridinyl-substituted quinoline was achieved through bismuth nitrate-catalyzed Diels-Alder reaction [4d]. Bismuth nitrate-catalyzed microwave-mediated route for the synthesis of dihydropyridines was realized [4e]. Octahydroxanthenes were prepared through bismuth iodide-catalyzed microwave-induced reaction [4f]. Ammonium chloride was found to be an excellent choice for the preparation of diverse pyrroles under microwave-induced conditions [4g, 4h].

Synthesis of Indoles by Microwave-induced Reactions:

Bismuth nitrate was found to be an excellent catalyst for the electrophilic substitution reaction of 7-aza-indole with activated carbonyl compounds under microwave-mediated conditions [5a]. This reaction was slow because of the less reactivity of the 7-aza-indole system. In contrast, the same reaction with indoles proceeded fast [5b]. Microwave-induced ruthenium chloride-catalyzed reaction of isatin with 4-hydroxy proline derivatives produced 3-pyrrple substituted indoles in excellent yield [5c].

Solventless Reactions:

Reactions described above were also performed in the absence of solvents. It was gratifying to note that these reactions are completed within a few minutes instead of hours that are required in the presence of solvents. Iodine-catalyzed Michael reactions of indoles with unsaturated carbonyl compounds were performed under solventless conditions [6a]. Bismuth nitrate-catalyzed synthesis of several *bis* indole derivatives were prepared in excellent yields [6b]. A facile synthesis of N-substituted pyrrole derivatives were conducted *via* bismuth nitrate-catalyzed reaction in the absence of any solvent [6c]. These pyrrole syntheses were also performed by iodine-catalyzed reaction in the absence of solvent [6d]. Polyhydroquinoline was prepared in the absence of solvent *via* microwave-induced reaction [6e]. Pechmann reaction was performed in the absence of any solvent *via* bismuth nitrate-catalyzed microwave-mediated reaction [6f]. Bismuth nitrate-catalyzed microwave-induced simple enamination of β-dicarbonyl compounds was performed in the absence of solvent [6g].

Reactions in Water:

Many organic reactions were performed in water. For example, bismuth nitrate-catalyzed Michael reaction of indoles with unsaturated ketones was conducted successfully in water [7a]. Aza-Michael reaction was also done in water [7b]. Dowex-50 was found to catalyze synthesis of dihydropyrimidines in water [7c]. Indium metal-catalyzed Strecker reaction of imines was

conducted in water successfully [7d]. Phosphoric acid-catalyzed Aza-Michael reaction was performed in water [7e]. This reaction was also conducted in water in the presence of ultrasound [7f]. Diverse pyrroles were prepared following Paal-Knorr method in aqueous carbon dioxide [7g, 7h].

One-Pot method:

Synthesis of new dibenzofluorene derivatives was performed in a one-pot operation. Thus, reaction of 2-naphthylbromide with beta tetralone in the presence of sodium hydride afforded the alkylated ketone. This ketone on cyclodehydration and aromatization reaction in a one-pot method produced dibenzofluorene in good yield. An extension with methoxy aromatic bromide was also possible for the preparation of penatcyclic derivative. These hydrocarbons were converted to numerous compounds that have demonstrated promising anticancer activities [8]

Various Reactions:

Our group had also investigated numerous reactions under green chemistry conditions. For example, bismuth nitrate-mediated microwave-induced selective hydrolysis of amide was performed [9a]. Diels Alder reaction was investigated under an identical condition with great success [9b]. N-bromosuccinimide and polystrenesulfonate-catalyzed microwave-assisted synthesis of various pyrroles were achieved [9c, 9d, 9e]. Microwave-assisted bismuth nitrate-induced oxidation of curcumin to vanillin was performed [9f]. Aza-Diels Alder reaction proceeded in the presence of bismuth nitrate and microwave irradiation [9g].

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