Studies of Microwave-assisted Eco-Friendly Synthesis of Antimicrobial And Antifungal Evaluation of Microcyclic Complexes.

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ABSTRACT

On the basis of recent publications to the use of microwave irradiation in synthesis of different area of chemistry. It was clear that this methodology would have a marked influence in all area of this synthesis chemistry. Uses of reduction reaction improved yields, modifications of selectivity, increased product purities and simplification of work-up procedures were described and, in most cases, these conditions and results could not be achieved by classical heating because many other factors affected the rate of chemical reactions. This methodology can be included within the concept of Green Chemistry because the strong absorption of microwave irradiation by the component of the reactants materials would lead to shorter reaction time interval with improved energy efficiency therefore the rate of chemical reaction increase. Moreover, the energy with solvent-free conditions, solid catalysts and green solvents has expanded the green applications of this non-conventional energy source which decrease activation energy with increase the rate of chemical reactions. Finally, the use of flow systems, another green methodology, has been used to permit the scale-up of microwave-assisted reactions under green conditions in various different areas of synthesis reaction. At present years Green chemistry is import tools to use the different area of synthesis reactions.

Introduction

Due to the ability of some compounds solids or liquids to transform electromagnetic energy into heat, microwave radiation has been widely employed in chemistry as an energy source. Microwave irradiation has several advantages over conventional heating and these include homogeneous and rapid heating spectacular accelerations in reactions as a result of the heating rate which frequently cannot be reproduced by classical heating and selective heating.[1-5] Consequently, microwave-assisted organic reactions produce high yields at low temperature and gives lower quantities of by products, while the purification of products is easier and, in some cases, selectivity is modified to use the selected product in same condition of reactants. Indeed, new reactions and conditions that cannot be achieved by conventional heating can be performed using microwaves with respected to normal methods. The use of microwaves in organic synthesis has been reviewed in numerous recent publications and other area of chemical reactions and reviews. Absorption and transmission of microwave energy is completely different from conventional heating Conventional heating is a superficial heating process and the energy is transferred from the surface of one system to the bulk of other system by convection and conduction.[6-10] This is an inefficient mode of heating because the surface is at a higher temperature than the bulk and the vessel must be overheated to achieve the desired temperature to the suitable chemical reactions. In contrast, microwave irradiation produces efficient internal heating by direct coupling of microwave energy with the bulk reaction mixture of different types of chemical reactions.[11-13] The magnitude of the energy transfer depends on the dielectric properties of the molecules. As a guide, compounds with high dielectric constants tend to absorb microwave energy whereas less polar substances and highly ordered crystalline materials are poor absorbers with bad conductor of electricity.[14-15] In this way, absorption of the radiation and heating can be very selective in the synthesis of different types of chemical reactions

Materials and Methods

Melting points of complexes were determined by open glass capillary method. All chemicals used were AR grade reage and were dry before used. A Laboratory Microwave Oven operating at 2450 MHz and power output of 600 W was used for all the experiments. The completion of the reactions was monitored by TLC. IR spectra were recorded on a Shimadzu FTIR-420 spectrophotometer. 1H NMR and 13C NMR spectra were recorded at 400°C on a Bruker AVANCE DPX (400 MHz) FT spectrometer in $CDCl_3$ using TMS as an internal reference. Mass spectra were recorded on JEOL SX-303 mass spectrophotometer at 70ev. Elemental analyses were carried out using a Coleman automatic C, H, N analyzer.

MICROWAVE ASSISTED SYNTHESIS OF COMPLEXES

Propane-1,3-diamide and ethanedioic acid was of AR grade and were distilled then dried before use in laboratory. While metal salts were of BDH quality. The synthesis of ligand by the chemical reaction between Propane-1,3-diamide with ethanedioic acid mixture was refluxed in water bath for 3 hour. The solid which separated at the end of the refluxing period was filtered then dissolved in boiling water and purified to give colourless solid. H, C and N were estimated by semi-micro combustion method on a Carbo-Erba 1106 elemental analyzer. Metal were estimated using standard procedure. The IR spectra were recorded with Backmann IR-20 spectrophotometer in KBr disc.

The metal (II) complexes were prepared and characterized to undergo condensation reaction between ethyl alcohol solution of the ligand was mixed with ethyl alcohol solution of corresponding metal salts ions in an inert atmosphere of nitrogen while the reaction between them is quite facile and the resulting mixture refluxed for 3-4 hour in water bath then on cooling the resulting complexes were obtained as colored crystalline solid. The reaction occurs in the present of catalyst $AlCl_3$

Results and Discussion

The conventional method available for the preparation of different compounds are not satisfactory because it requires long reaction times interval and its use are highly expensive and hazardous solvents and yield are only moderates.

The reactions were also carried out using a thermo stated oil-bath at the same temperature (120^{o}C) as for the MW activated method but for a longer interval of time. It was found that MW method has more suitable therefore it improved the yields significantly during the chemical. This observation can be rationalized on the basis of the formation of dipolar activated complex from uncharged adduct in these reactions and greater stabilization of the more dipolar activated complex by dipole-dipole interaction with electric field of the microwaves as compared to the less polar adduct which may reduce the activation energy resulting in the rate enhancement.

The electron donating groups in the complexes of the system plays a significant role in the bioactivities. In addition to this, the presence of heteroatom such as oxygen, and nitrogen also play a vital role in the observed antibacterial and antifungal activity. It is also suggested that the nitrogen containing compounds might inhibit enzyme synthesis, since enzymes need specific groups for their activity and are especially susceptible to deactivation by the compounds. The presence of nitrogen atoms in the structure of the active compounds facilitates their diffusion through the lipid layer of the microorganism membranes to the site of action, eventually killing them or control by linking with essential groups of certain cell enzymes. The electrochemical properties of the microcyclic complexes were investigating polarographically in DMF water medium in presence of sodium perchlorate as supporting electrolyte. Polarographic measurements were caused out with reference to saturated calomel electrode. All the complexes are found to undergo one step reduction reversibly or quasi reversibly and the redox reaction step is accompanied by one electron transition. The thermodynamics parameters $E_{1/2}$ for the complexes are found within the region -1.35 to -1.6 Dv. It is interesting to note that complexes with partial or complete conjugation in the microcyclic framework.

Antimicrobial and antifungal activity

Fungicidal activity of the complexes was done by disc plate method on Pencillium expanse and as perilous flatus. On the basis of comparison with reference to fungicide, the complexes were found to be more effective than due to chelation theory.[16-18] it seems that enhanced antifungal and antimicrobial activity for the compound is due to its electron donating group and the poly-conjugated nature of the compound with central ion of the complexes. On the basis of conjugation and donating effect the compounds provide large surface areas which enhance greater extent lipophilic and absorbing nature. The complexes are more active due to the greater dissolving ability in fats, oils, lipids and non-polar suitable organic solvent such as hexane, toluene, benzene … etc with more absorbing nature of the complexes with respect to free metal ions, which controls the growth or increases the antifungal and antimicrobial activity.[19-20]

Conclusion

The major challenges for medicinal chemistry with other area of medicines in all over the world to the development of simple, suitable, with more effective and eco- friendly method for the synthesis of different types of compound which enhance antimicrobial and antifungal activity. Now a present day various methods in area of medicinal chemistry have been proposed for the preparation of compounds and their derivative but, all of this reported method has several drawbacks like ,use of organic solvents which causes, pollution, under drastic conditions reaction, time consuming reactions and use of expensive reagents therefore the products are highly expensive. To solve such type of problems here we report a simple, practically feasible and eco-friendly method for the synthesis of complexes with its derivatives. The growth of green chemistry is important because it has many advantages like, short reaction time interval, eco-friendly, and pollution free or generates very less pollution and consume low amount of energy. In view of importance of compounds and their derivatives as a chemotherapeutic agent, and development of a facile, efficient and environmentally benign method to synthesize these different types of compounds would be great cost value. This observation prompted us to develop an environment friendly approach for synthesis of substituted suitable complexes which possesses various antimicrobial and antifungal activities in different area.

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