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NOVEL GREEN AND CONVENTIONAL TACTIC IN THE ROUTE OF AMALGAMATION OF 1-PHENYL NAPHTHALENE LIGNAN

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Abstract
In recent study Zeolite has remarkable uses for their applications in organic synthesis. Green approach towards synthesis of 1-phenyl naphthalene and their derivatives from β benzoyl propionic acid (βBPA) in few segments. Precursor βBPA prepared by fridel craft reaction by using green reagent i.e. Zeolite. In initial segment, comparative study of synthesis of α-arylidine γ-phenyl δ,β- butenolide by perkin condensation reaction using Zeolite with different catalyst. Second segment synthesis of α-arylidine, β-Benzoyl Propionic acid by cleavage using alcoholic sodium carbonate. In terminating segment 1-phenyl naphthalene has been already synthesized by cyclizations using PPA, H2SO4, sulphamic acid and nanozeolite has been introduced as a cyclizing reagent in place of foresaid. It has been observed that microwave method creates better yield, reduces reaction time and energy compared with conventional synthesis lignans.

Keywords*: 1-phenyl naphthalene, α-arylidine γ-phenyl δ,β- butenolide*, *nanozeolite.*

1. Introductions

1.1 Green Chemistry

During the early 1990s the US Environmental Protection Agency (EPA) was coined the phrase 'Green Chemistry' by Prof. Anastas to promote innovative chemical technologies that reduce or eliminate the use of generation of hazardous substances in the assign manufacture and use of chemical products. It is widely acknowledged that there is a growing need for more environmentally acceptable processes in the chemical industry (1-4). This trend towards what has become known as Green Chemistry or sustainable technology necessitates a paradigm shift from traditional concept of process efficiency, that focus largely on chemical yield and to one that assigns economic value.

Green chemistry is an essential chemistry for the environment. The term green chemistry is defined as the invention, design, development and application of chemical products and process to reduce or to eliminate the use and generation of substance which are hazardous to human health and the environment. It is the chemistry which is being by design. Such green chemistry is governed by twelve principles that any chemist should consider in planning synthesis some of this concept include the minimal use of hazardous reagent or reactant, the prevention of waste and by product design and utilization of less hazardous. Safer chemicals and chemical of auxiliaries, energy, efficiency, use of renewable resources, a high degree of incorporation reactants into the desired target, and a heavy emphasis on the use of catalytic process; in other works for sustainable solutions in chemical industry.

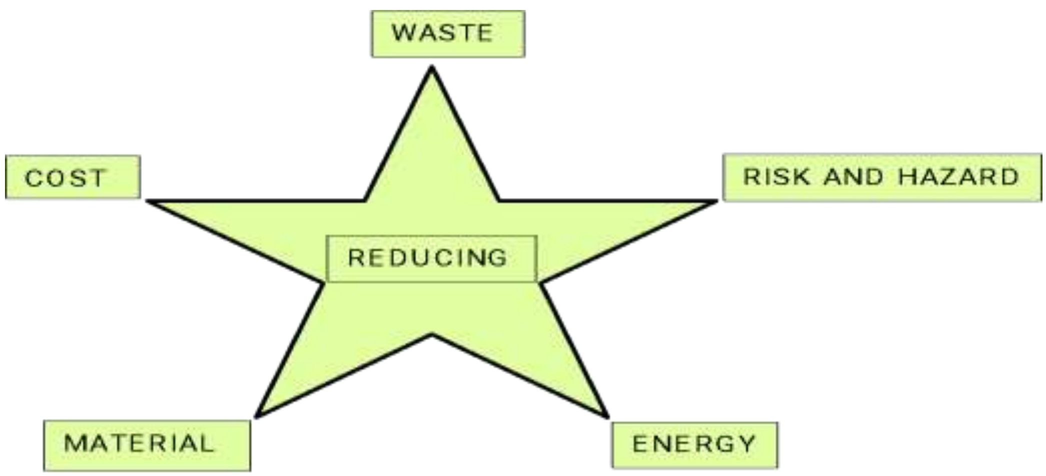

Fig. 1 Green Chemistry for reduction Process

1.2 ATOM ECONOMY

Stanford chemistry Professor Barry Toast, who first proposed the concept of atom economy in 1991 (5-7). The atom economy of process is calculated by dividing the molecular weight of the desired product by the sum of molecular weight of all the substances produced in stoichiometric equation.

In his 1991 publication, Trost challenged chemist to pursue greener chemistry but making synthesis more syntheses more atonomically economical.

1.3 PRINCIPLES OF GREEN CHEMISTRY

I. Waste prevention instead of remediation:

This principle is the most obvious and over arches the other principles. It goes back to the old age “an ounce of prevention is worth a pound of cure.” It is better to prevent waste than clean it up after the fact.

II. Atom Economy:

This principle gets into the actual chemistry of how products are made. This principle states that it is best to use all the atoms in a process. And, those atoms that are not used end up as waste. The atom economy is a simple calculation that can be used when teaching stoichiometry and chemical reactions. The calculation is: A.E. = formula weight of product divided by the formula weight of all the reactants. It is a simple measure of the amount of waste in a process.

III. Less hazardous/toxic chemicals:

This principle is focused on how we make molecules and materials. The goal is to reduce the hazard of the chemicals that are used to make a product (the reagent). Throughout the history of how we have invented products and developed the process for making them, chemists have traditionally not thought about what reagents they are using and the hazards that are associated with them. Chemists have traditionally used whatever means necessary. Today we are finding that less hazardous reagents and chemicals can be used in a process to make products and, many times they are made in a more efficient manner.

IV. Safer products by design:

The previous principle was focused on the process. This principle focuses on the product that is made. Everyone wants safe products. Everyone also wants products that so what they are supposed to do (they have to work). This principle is aimed at designing products that are safe, non-toxic and efficacious. A good example of this is pesticides; which are products that are designed to be toxic. Many researchers are focused on created pesticides that are highly specific to the pest organism, but non-toxic to the surrounding wildlife and ecosystems.

V. Innocuous solvents and auxiliaries:

Many chemical reactions are done in the solvent. Traditionally, the organic solvent have been used that possess hazards and many are highly toxic. They also create volatile organic compounds (VOC's) which add to pollutants and can be highly hazardous to humans. This principle focusing on the creation of product in such a way that they use less hazardous solvents. We use solvents in our daily life and in chemistry laboratory.

VI. Energy efficient by design:

There is a focus on renewable energy and energy conservation. We use energy for the transportation purpose and to provide electricity to our homes and business. Traditional method for generating electricity has been found to contribute to global environmental problems such as Global Warming and energy used can also be of a significant cost. This principle focuses on creating products and material in a highly efficient manner and reducing the energy associated with creation of products. Thus reducing associated pollution and cost.

VII. Preferably renewable raw material:

The products 90-95% of we use in our daily life are made from petroleum. Our society not only depends upon petroleum for transportation and energy but also for making products. These 4 principles are sought to shift our dependence on petroleum and to make products from renewable materials that can be gathered or harvested locally. Biodiesel is one of the examples of this where researchers are trying to find alternative fuel s that can be used for transportation. Another example is bio based plastic. PLA (Poly Lactic Acid) is one plastic that is made from renewable feed stock such as corn and potato waste.

VIII. Shorter Synthesis (avoid derivatization):

This principle is perhaps most abstract principle for non-chemist. The methods that chemists used to make the products are sometimes highly sophisticated. Many involve the manipulation of molecules in order to shape the molecule in shape that we want to look them like. This principle aims to simplify that process and to look at natural system in order to design the products in the simplified manner.

IX. Catalytic rather than stoichiometric reagents:

In a chemical process catalysts are used in order to reduce energy requirements and to make happen the reaction more efficiently. Another benefit to use a catalyst is small quantity reactants requirement. If the catalyst is truly green then it should not been toxic and can be used over and over. Chemists are investigating use of enzymes to perform chemistry in laboratory in order to obtain desired product.

X. Design product for degradation:

Not only we do want material and products to come from renewable sources but, we would also like them to not persist in the environment. There is no question that many products we use in our daily life are far too persistent. Plastics don't degrade in our landfill and pharmaceutical drugs such as antibiotics build up in our waste water system. This principle seeks to design the product in such a way that they perform their intended function and then, when appropriate, will degrade into safe, innocuous byproducts when they are disposed off.

XI. Analytical methodologies for pollution Prevention:

According to this principle, analytical methodologies need to be further developing to allow for real time in process monitoring and control to prior the formation of hazardous substances.

XII. Inherently safer Processes:

This principle focuses on the safety for the worker and the surrounding community where an industry resides. It is better to use the material and the chemicals that will not explode, light on fire, ignite in air, etc. when making a product. There are many examples where safe chemicals are not used and the result was disaster. The most widely known and perhaps one of the most devastating disaster was that of Bhopal, India in 1984where a chemical plant had an accidental release that resulted in thousands of lives had lost and many more long lasting injuries. The chemical reaction that occurred was an exothermic reaction that went astray and toxic fumes were released to the surrounding community. When creating the products, it is best to avoid the highly relative chemicals that have potential to reduce in the accidents. When explosions and fires are happened in the industries, the result is often divesting.

One of the ways of implementing the principle of the green chemistry is to use catalyst. It plays a significant role in reducing pollution in our environment. With it reaction can be more efficient and selective thereby eliminating large amount of byproduct and other waste compounds. Catalysts has a crucial importance for chemical industries and it is use to make an enormous range of products like heavy commodity and fine chemicals. It is one of the fundamental pillars of green chemistry and is considered as the most preferred and relevant technology to achieve a reduction in waste from chemical process by use of cleaner synthetic methods.

1.2 Catalysis:

Catalyst accelerates reactions by orders of magnitude, enabling them to be carried out under the most favorable thermodynamic regime and at much lower temperature and pressures. Thus catalyst is the key factors in reducing both the investment and operation costs of chemical process (8-9). The chemical industry of the 20th century could not have developed to its present status on the basis of non-catalytic, stoichiometric reactions alone. Reactions can in general be controlled on the basis of temperature, concentration, and pressure and contact time. Raising the temperature and pressure will enable stoichiometric reaction to proceed at a reasonable rate of production but the reactors in which such conditions can be safely maintained become progressively more expensive and difficult to make. Acid catalysis is by far the most important area of liquid seven phase industrial reactions depend on the use of inorganic or mineral acids, while many of these processes are catalytic, some require (e.g. acylation using anhyd $AlCl_3$) stoichiometric amounts of acids. Some of the major reaction types which are important in this context are friedel craft alkylations, acylation and sulphonylatons, aromatic halogenations, nitrations, isomerixzations and oligomerizatins. These reactions are genrally catalyzed by mineral acids such as H_2SO_4 and HF: and by lewis acids such as $AlCl_3$ and BF_3 (10-13). These reagents are hazardous in handling, damaging the plant through their corrosiveness and add process difficulties through the use of quenching and separation stages which led to large volume of toxic and corrosive wastes. These acids such as H_2SO_4, HF, $AlCl_3$ and BF_3 are typically soluble in organic reaction medium or remain as a separate liquid phase. at the end of the reaction, such acid are normally destroyed I water quenching stage and require subsequent neutralization: thus consuming additional (alkaline) resources and producing salt waste. For their alternative process we use new green catalyst as Zeolites [14-16].

1.3 Ideal Synthesis:

Paul Wender introduced the concept of ideal synthesis in view to make the chemical process green and clean (17-20). Ideally one would seek to design a synthesis in which target molecule is made from readily available starting material in one simple, safe, environmentally acceptable and resources effective operation that proceeds quickly and in qualitative yield.

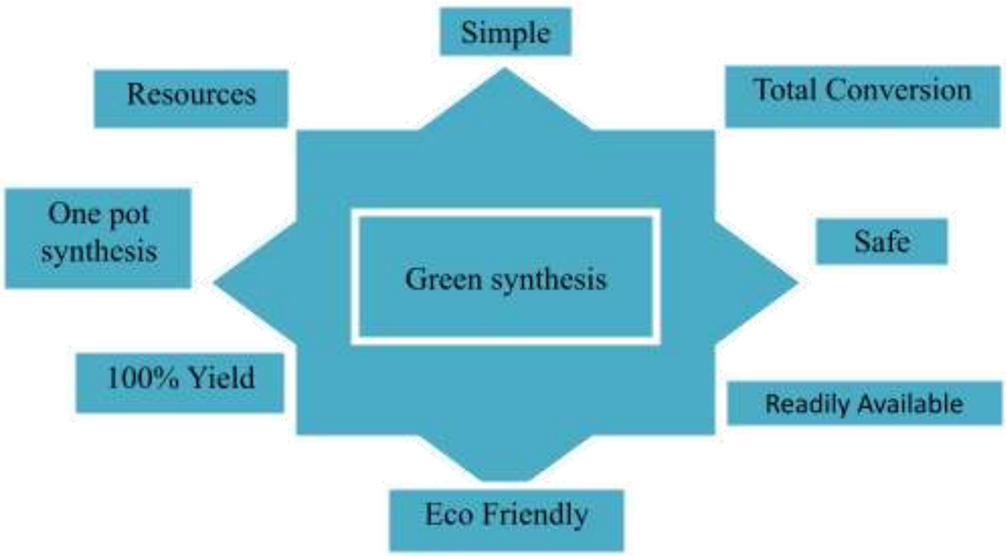

Fig. 2: Ideal Synthesis in terms of green chemistry

2. Experimental (Green source of Energy)

In 1855, Robert Bunsen invented the burner which act as energy source for heating a reaction vessel; this was suspended by isomental energy required to bring about chemical reaction is supplied largely by external sources of heat such as steam, oil bath and electrical heating elements. There is currently growing interest in alternative or green sources of energy that can target specific molecules or bonds, giving both energy savings and improved selectivity. One of the sources of energy is the Bunsen burner of the 21st century such as Ultrasonication oven.

2.1 Ultrasonications:

Sonication is defined as the process in which sound waves are used for agitating the particles in the solutions. These disruptions are used for mixing of the solutions, to increase the speed of dissolution of a solid into a liquid, and for the removal of dissolved gases from the liquids. Ultrasonication technology has been used in chemistry since the late 1970s but it has only been implemented in organic synthesis since the mid of 1980s. Ultrasonication is a form of electromagnetic radiation that falls at the lower frequency rage.

2.2 Principle:

The basic principle behind the heating in Ultrasonication oven is due to the interaction of charged particle of the reaction material with electromagnetic wavelength of particular frequency. The phenomena of producing heat by electromagnetic irradiation are ether by collision or by negative with each cycle of the wave. This case rapid orientation and reorientation of travel through the material (e.g. Electron in a sample of carbon) a current will induce which will travel in phase with the field. If charge particle are bound within regions of the material, the electric field component will cause them to move until opposing force balancing the electric force. In the ultra-sonication process, cavitation leads to dispersion, homogenization, disintegration, emulsions, extraction, and sonochemical effects of the liquids. High power ultrasound is introduced to the liquid which creates regions of high pressure (known as compression) and low pressure (known as rarefaction). The creation of these regions is dependent on the rate of frequency at which the ultrasound is applied. When low pressure is applied to the liquid, high-intensity ultrasonic waves are produced, creating small vacuum bubbles in the liquid. As the bubbles reach their saturation level, they collapse and this happens in the high-pressure cycle. This process is termed cavitation. During cavitation, the bubbles in the liquid can jet up to 280 m/s velocities. The below figure explains how the sound wave propagates in the liquid resulting in the formation of bubbles and their collapse.

2.3 Ultrasonication frequency:

Ultrasonication heating refers the use of electromagnetic waves ranges from 0.01 m to 1 m wave length of certain frequency to generate heat in the material. These Ultrasonications lie in the region of the electromagnetic spectrum between millimeter wave and radio wave such as between Infra Red and radio waves. They are defined as those waves with wavelength between 0.01 meter to 1 meter, corresponding to frequency of 30GHz to 0.3 GHz.

2.4 Ultrasonication Assisted organic synthesis (UAOS):

Synthesis of new chemical entities is major bottleneck in drug disacovery. Conventional methods for various chemical syntheses are well documented and practiced. The methods for synthesis (heating process) of organc compounds have continuously modified from the decade. Burner which acts as energy source for heating reaction vessel: this was later superseded by is mental, oil bath or hot plate, but the drawback of heating though method remains the same. Ultrasonication assisted organic synthesis (UAOS), which has developed in recent years, has been considered superior to traditional heating [21-22]. UAOS has emerged as new “lead” in organic synthesis. The technique offers simple, clean, fast, efficient and economic for the synthesis of large number of organic compounds. In the recent year UAOS has emerged as new tool in organic synthesis. Important advantages of this technology include highly accelerated rate of reaction, reduction in reaction time with an improvement in the yield and quantity of the product. Now day’s technique Is considered as an important approach towards green chemistry, because this technique is more environmentally friendly. This technology is still under-used in the laboratory and has the potential to have impact on the fields of screening, combinatorial chemistry, medicinal chemistry and drug development. Conventional method of organic synthesis usually need longer heating time, tedious apparatus setup, which result in higher cost of process and the excessive use of solvents reagents lead to environmental pollution. This growth of green chemistry holds significant potential for a reduction of the by product, a reduction in waste production and a lowering of the energy costs [23-25]. Due to its ability to couple directly with the reaction molecule and by passing thermal conductivity leading to a rapid rise in the temperature Ultrasonication irradiation has been used to improve many organic synthesis methods.

2.5 Ultrasonication Heating Mechanism:

Sonication is the act of applying sound energy to agitate particles in a sample, for various purposes such as the extraction of multiple compounds from plants, microalgae and seaweeds. Ultrasonic frequencies (> 20 kHz) are usually used, leading to the process also being known as ultrasonication or ultra-sonication.

In the laboratory, it is usually applied using an ultrasonic bath or an ultrasonic probe, colloquially known as a sonicator. In a paper machine, an ultrasonic foil can distribute cellulose fibres more uniformly and strengthen the paper.

Ultrasonication is unique homogenization technique utilized in variety of applications. It is processes which break large particle into smaller fragment or better uniform sized particles in the base fluid. Sonication of nanofluid is achieved by providing sound energy to agitate the nanoparticles in the suspension. Ultrasonication is a process where above 20 kHz of ultrasonic rates/frequencies are utilized for homogenization. Two commonly equipped sonicators are Bath-type and probe-type. Probe type sonicators having high intensity is noticed to be more effective than bath-type.

The sonication process uses ultrasonic sound waves. During the process, there is a production of thousands of microscopic vacuum bubbles in the solution due to applied pressure. The formed bubbles collapse into the solution during the process of cavitation. The collapsing of bubbles takes place in the cavitation field leading to the generation of enormous energy as there is a production of waves. This results in the disruption of the molecular interactions between the molecules of water. As there is a reduction in the molecular interactions, the particles start to separate and allow the mixing process to take place. There is a release of energy from the sound waves that result in friction in the solution. Ice cubes are used during and after the sonication process to prevent the sample from heating up.

2.4.6 Parts of Sonicator

The equipment used for sonication is known as a sonicator. The following are the three parts of the sonicator:

- Generator
- Transducer
- Probe

The generator is used for transforming the input electrical power into an electrical signal that drives the transducer.

The transducer is used for converting the electrical signal into vibration. This vibration is used in the probe tip by amplifying it into a longitudinal vibration causing a cavity in the sample. The ultrasound energy is the creation of cavitation which causes the disruption of the sample and makes it easy to break down the particles into smaller ones.

2.7 Equipment:

Substantial intensity of ultrasound and high ultrasonic vibration amplitudes are required for many processing applications, such as nano-crystallization, nano-emulsification. Deagglomeration, extraction, cell disruption, as well as many others. Commonly, a process is first tested on a laboratory scale to prove feasibility and establish some of the required ultrasonic exposure parameters. After this phase is complete, the process is transferred to a pilot (bench) scale for flow-through pre-production optimization and then to an industrial scale for continuous production. During these scale-up steps, it is essential to make sure that all local exposure conditions (ultrasonic amplitude, cavitation intensity, time spent in the active cavitation zone, etc.) stay the same. If this condition is met, the quality of the final product remains at the optimized level, while the productivity is increased by a predictable "scale-up factor". The productivity increase results from the fact that laboratory, bench and industrial-scale ultrasonic processor systems incorporate progressively larger ultrasonic horns, able to generate progressively larger high-intensity cavitation zones and, therefore, to process more material per unit of time. This is called "direct scalability". It is important to point out that increasing the power capacity of the ultrasonic processor alone does not result in direct scalability, since it may be (and frequently is) accompanied by a reduction in the ultrasonic amplitude and cavitation intensity. During direct scale-up, all processing conditions must be maintained, while the power rating of the equipment is increased in order to enable the operation of a larger ultrasonic hornFinding the optimum operation condition for this equipment is a challenge for process engineers and needs deep knowledge about side effects of ultrasonic processors

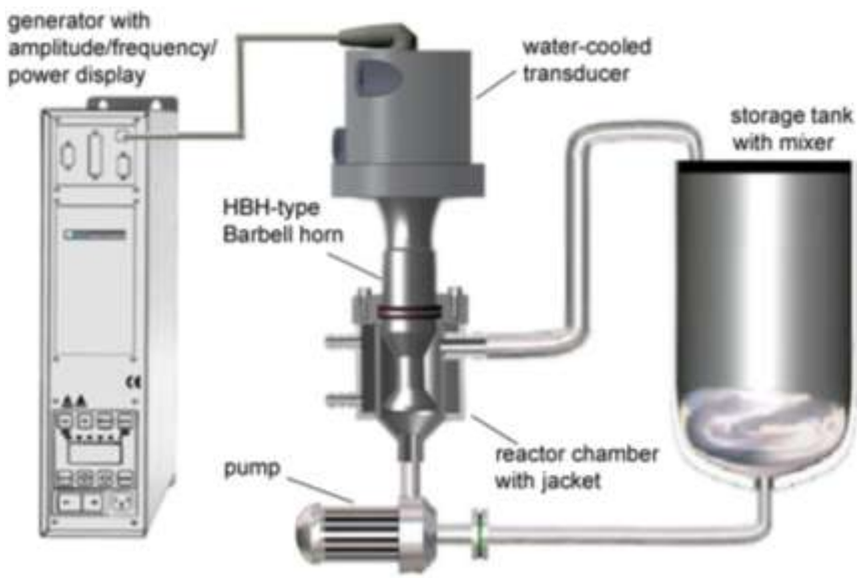

Fig. 3 Schematic of lab-scale ultrasonic liquid processors

3. Sonication Methods:

There are two sonication methods and they are:

3.1 Direct sonication method

In the direct sonication method, the probe is directly inserted into the sample, and it is the most common method of sonication. In this method, the energy is transmitted from the probe to the sample directly. This is a high-intensity process and therefore, the processing of the sample takes place quickly. The determination of the volume of the liquid for processing is done by studying the diameter of the probe’s tip. These tips are either replaceable or solid tips made from titanium.The smaller the diameter of the tip, the higher is the intensity of sonication and the energy is confined to smaller areas. As the diameter of the tip increases, the processing volume becomes larger, however, the intensity becomes lower. The output of the large-diameter probes can be increased by using boosters and high gain horns.

3.2 Indirect Sonication Method

In the indirect sonication method, the contact between the probe and the sample is eliminated. This method is also known as the high-intensity ultrasonic bath. The ultrasonic energy is transmitted from the horn to different tubes of the sample through the water. This method is preferred when the volume of the sample is low as it reduces the foaming and loss of the sample.The indirect sonication method finds application in pathogenic or sterile samples as this method prevents the contamination of the samples. The cup horn and the microplate horn are the two indirect sonicators that are considered ideal for throughput applications.

3.3. Application in organic synthesis

The sonication mechanism is used in ultrasonic cleaning which includes cleaning of particles that adhere to the surfaces.It is used in laboratories for cleaning fragile objects such as spectacles and jewellery.The artificial ageing of liquors and other alcoholic beverages is done by the process of sonication.Other applications of sonication in food industries include dispersions of emulgators and speeding the filtration process.The below is an explanation of the characteristics of sound waves:for example, will have a stronger dipole to cause more rotational movement in an effort to align with the changing field.

3.4. Effect of Solvent

A compound that is less polar, however, will not be as disturbed by the changes of the field and therefore, will not absorb as much Ultrasonication energy. Unfortunately, the polarity of the solvent is not the only factor in determining the true absorbance of Ultrasonication energy, but it does provide a good frame of reference. Most organic solvents can be broken into three different categories: low, medium or high absorber as shown in table 1. The low absorbers are generally hydrocarbons while absorbers are more polar compounds, such as most alcohols.

Table 1: Most organic solvents in different categories: low, medium or high absorber

Absorbance level	Solvents
High	DMSO, EtOH, MeOH, propanols, nitrobenzene, formic acid, ethylene glycols.
Medium	Water, DMF, butanol, acetonitrile, Methyl ethyl ketone, acetone, nitromethane, dichlorobenzene, 1,2 dichloroethane, acetic acid, trifluroacetic acid.
Low	Chloroform, DCM, cabon tetrachloride, 1,4 –dioxane, ethyl acetate, pyridine, triethylamine, toluene, benzene, chlorobenzene, pentane, n-hexane and other hydrocarbons.

4. Result and Discussion

4.1 Differentiation between conventional and Ultrasonication heating

Ultrasonication heating is different form of conventional hating in many aspects. The mechanism behind synthesis is quite different from conventional synthesis.

4.2 Green approaches towards the synthesis of 1-phenyl Naphthalene Lignan:

1-phenyl naphthalene has been the subject of great inter, as it is an important intermediate for synthesis of cyclo-lignans and also for their physiological properties.

Various attempts have been made for the synthesis of 1-phenyl naphthalene and the different types of lignans by large number of workers but only few were successful in synthesizing the naturally occurring isomers.

Green approach towards synthesis of 1-phenyl naphthalene involves key precursor like beta benzoyl propionic acid. The beta benzyl propionic acid have been synthesize via friedel craft reaction by many ways like general method like benzene, succinic anhydride and $AlCl_3$. It is replace by green methods as Ultrasonication using $AlCl_3$, conventionally Nanozeoliteand Ultrasonication using Nano catalyst.

In one of the several methods used for the synthesis of 1-phenyl naphthalene type of lignan, Haworth and co-workers prepared the system in a series of steps by starting with beta-benzoyl propionic aid. $Borsche_{50}$ had observed the remarkable properties of Beta-benzoyl propionic acid, having two reactive methylenes it underwent perkin condensation, which had been known mainly in the alkyl acetic acid and aryl acetic acid the yield 90% were surprisingly high. It was also noted that when the corresponding acid/ester was treated with aryl aldehyde it gave product which was later considered as inclusive in stobbe type of reaction. Perkin reaction was investigated in detail as a prelate to the synthetic development of system. It was thus envisaged to use the Beta- benzoyl propionic acid system to construct perkin condensation of the former with aryl aldehyde would yield Butenolide of the type, which ultimately would give way to alpha arylidine beta benzoyl propionic acid, latter under goes cyclization with difference cyclizing regents like zeolite, PPA, sulphamic acid, etc. to get 1-phenl naphthalene derivative.

It has reported that for the system of above type to undergo cyclization, the presence of hydroxyl, methoxy or methylenedioxy group at position para to the cyclization site in the aromatic ring is essential. Therefore, the reactions are restricted to aldehyde possessing such groups.

4.3 Conventional heating:

Reaction mixture heating proceeds from a surface of reaction vessels. This vessel should be in physical contact with surface sources that is at a higher temperature source (e.g. mental oil bath, steam bath, sand bath, etc.). Heating take place by thermal or electric sources involve in conduction mechanism. Transfer of energy occurs from the wall surface of vessel, to the reaction mixture and eventually to reacting species. In conventional heating, the highest temperature (for an open vessels) that can be achieved is limited by boiling of particular mixture. In conventional heating all the compound are heated equally and heating rate is less.

4.4 Ultrasonication Heating:

Reaction mixture heating proceeds directly inside mixture. No need of physical contact of reaction with higher temperature source while vessel is kept in Ultrasonication cavities. Heating take place by electromagnetic sources involve in dielectric polarization and conduction mechanism. The core mixture is heated directly while surface (vessel wall) is source of loss of heat. In Ultrasonication heating, the temperature of mixture can be raised more than its boiling point such as superheating take place. In Ultrasonication heating specific component can be heated specifically and heating rate is several folds high.

5. Conclusion :

The creation of environmentally friendly organic reactions has emerged as a critical and challenging study subject in current organic chemistry research as a result of the enormous environmental degradation brought on by the diurnal industrialisation of today. In this context, the current study intends to synthesise and their derivatives employing ultrasonication, irradiation, and Nanozeolitecatalyst in green ways; hence, the drive towards green chemistry. The use of Ultrasonication irradiation may outperform conventional reaction conditions in several aspects, such as synthesis, easier workup, reduction of usual thermal degradation products and reduction of toxic and explosive quantities of solvents (green solvent), etc. We had treated the benzene ring with the succinic anhydride along with the use of $AlCl_3$ as catalyst. $AlCl_3$ acts as highly reactive hydrogen abstracter. Whole the reaction had taken place in the influence of high energy Ultrasonication region rather than using burner.

The heating with burner brought the distribution of heat energy into heating of reacting mixture containing vessel as well as the reaction mixture, whereas the Ultrasonication provides all the energy to the reaction mixture without wasting it in the unwanted things like heating of vessel.

Due to the investment of whole energy for the conversion of the moiety into product, the time required for it is eventually reduce by a very large number.

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