

COMPARATIVE STUDIES ON GREEN CHEMICAL SYNTHESIS AND CONVENTIONAL SYNTHESIS OF “ACETANILIDE & DIBENZALACETONE” AND ITS EVALUATION PARAMETERSP.Sathya sowmya¹, H.Aliya,

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The demand of green chemistry for applying in the pharmaceutical and the other chemical industries is increasingly vital due to the fact that our world faces the environmental challenges of the twenty-first century. US Environmental Protection Agency (EPA) has suggested green chemistry for innovative technologies that reduce toxic, undesired waste, and environmental impact. Green chemistry is thus getting grew as an open light to afford a huge scientific area. After EPA, 12 principles of green chemistry have been gotten more attention and these principles have been considered more seriously by pharmaceutical companies since 1998. Pharmaceutical companies declared that they should improve the environmental performance by utilizing green chemistry. Not only pharmaceutical companies but also the other chemical industries started to take a step for green chemistry due to its advantages such as decreasing of waste and cost. It is assumed that green chemistry can save the industry an estimated USD 65.5 billion by 2020 primarily by reducing manufacturing costs¹. If the processes can be implemented right, green chemistry can afford to reduce waste product and decrease the resources consumption. Green chemistry is needed for minimizing of some social risks and safety issues, as well.

In the past decade, some of the large pharmaceutical companies around the world have focused on using green chemistry processes for drug discovery, development, and manufacturing. These firms include Amgen, the Merck Group, Abbott, Eli Lilly, Johnson & Johnson, and Roche. Green chemistry has started to point three lines such as cost, mankind, and our planet. American Chemical Society's Green Chemistry Institute's Pharmaceutical Roundtable was therefore launched and, since 2008, many drug companies have become the members that aim to foster the development of more efficient, less polluting processes. Fortunately, green chemistry celebrates 25 years of progress on 2016³.

It cannot be denied that people need medicines to cure their diseases some of which are very unpleasant. For that reason, pharmaceutical industry has struggled to have modern synthetic strategies for known and unknown therapeutic reagents. On the other hand, although many successful methodologies were achieved, the toxic properties of many reagents and solvents were not known and the issues of waste minimization and sustainability of solvents and/or uncreated reagents were largely unheard⁴. Chemists and medicinal scientists can reduce the risk to human health and the environment by following all the valuable principles of green chemistry. The most simple and direct way to apply green chemistry in pharmaceuticals is to utilize eco-friendly, nonhazardous, reproducible, and efficient solvents and catalysts in the synthesis of drug molecules, and in researches involving synthetic chemistry.

It has become clear that the chemical and related industries such as pharmaceutical companies are faced with environmental problems. There are a lots of synthetic methodologies and they have generated abundant amounts of waste and chemical industries want to minimize or eliminate this waste. Sheldon has discussed that in the pharmaceutical industries, there is an urgency for consideration of the waste product as a number of by-products produced per kg of product (designated E factor). Innovative strategies on chemistry are the core of the pharmaceutical business. The main point is gathering technology and chemistry to improve lives of patients and minimize environmental impact.

Solvents and stoichiometric reagents are the most important parameters to be considered for greener strategies and these parameters are under investigation in detail by many pharmaceutical companies such as -Aventis and

GlaxoSmithKline. These companies have suggested that conventional solvents such as halogenated, petroleum-based should be converted into greener solvents such as glycerol, ethyl lactate, and water. A catalyst is another crucial parameter which reduce the amount of inorganic salts and/or reagents. Green alternative for consuming of stoichiometric salts and reagents is to use a catalyst and this issue has been considered by pharmaceutical companies. However, demanding of the least expensive reagents has limited the applying of catalysts to be used widely.

Future perspective of green chemistry will be extended more seriously in many research areas. Product and environment should be considered together and it should be remembered that this planet needs a balance of nature. Every attempt to heart this balance will come across more serious effects. That is why we need greener strategies and greener thinking.

Synthetic strategies with green solvent

In every product development processes and different industrial applications, solvents are needed in huge amounts resulting in abundant amounts of waste⁶. Innovative technologies and different synthetic strategies have discussed solventless methods which are not accepted for all areas of research due to some market concerns. After the solventless ideas, chemists and medicinal scientists have searched out for solvents which suit green chemistry. According to Fischer, green solvent expresses the target to minimize the environmental impact coming from the consuming of solvents in chemical production. Some strategies have emerged for solvents which can be mentioned as green. These are substitution of hazardous solvents resulting in more eco-friendly, biodegradable, and/or minimizing of ozone depletion potential, use of biosolvents (oleochemicals), and substitution of organic solvents which are supercritical fluids and ionic liquids¹⁰. In the literature, many examples of green solvents can be seen for forming natural products, medicines, and important intermediate products which can be used for further synthesis.

APPLICATIONS OF GREEN CHEMISTRY

Chemicals from glucose: These are the chemical compounds are a set of chemicals which might be made on a completely massive scale to satisfy international markets. Glucose is alternative for product chemicals. Biotechnological strategies are used to control the production of [fragrant compounds], compounds inclusive of catechol, hydroquinone, and adipic acid, every compounds of which be able to be vital, may be synthetic. Benzene is the initial material used for these materials, by means of changing benzene amid glucose can assist in lowering the usage of diverse reagents with certain toxic. Synthesis which takes region in water as a replacement for of natural solvents is more beneficial.

Polysaccharide Polymers: They are an essential group of compounds that include widespread packages. they have got their dangerous consequences. The big range of compounds can be exploited. Polysaccharide because the feedstock have to be used as beginning materials due to the fact that it's far extra environmentally feedstock. Those are organic and have the benefit of being renewable or viable, in place of petroleum feedstock. On the opposite side these don't have any chronic toxicity to environment and health of humans.

MATERIALS AND METHODS:

MATERIALS:

S.NO	EQUIPMENT	MODEL	DEPARTMENT
1.	UV spectrophotometer	SYSTRONIC Double beam spectrophotometer 2203	SKUCOPS
2.	water bath	VWR MODEL -1250	SKUCOPS
3.	Melting Point Apparatus	SICBMPA -01	SKUCOPS
4.	Weighing Balance	Model PG/FB,DS-852G	SKUCOPS

2.2:REQUIREMENTS:

- Aniline
- Glacial Acetic Acid

- Acetic Anhydride
- Zinc Dust
- Distilled Water
- Round Bottom Flask
- Funnel
- Reflex Condenser
- Stirrer
- Filter paper
- Tripod Stand
- Beaker
- Pipette

METHODS :

SYNTHESIS OF ACETANILIDE BY GREEN CHEMICAL SYNTHESIS:

- 1) Take 10 ml of Aniline,10ml of Glacial Acetic Acid and add 10ml of Acetic Anhydride add pinch of Zinc dust in a 100ml of Round Bottom Flask.
- 2) Fix a Reflex Condenser to the Round Bottom Flask.
- 3) Heat the mixture for 15-20min on a Water Bath.
- 4) Pour the hot mixture into a Ice Cold water with constant Stirring.
- 5) Filter the crude product by using the Filter Paper.
- 6) From the Crude Product get the Pure Crystals through Crystallization Process.
- 7) Take a beaker and add 20ml of water to the crude product.
- 8) And also add Activated Charcoal.
- 9) Heat the mixture for 10min.
- 10) Cool down the mixture to room temperature.
- 11) Filter the product and crystals from the solution

SYNTHESIS OF ACETANILIDE BY CONVENTIONAL METHOD:

- Take 10ml of Aniline add 20ml of Glacial Acetic acid and Acetic Anhydride in a beaker.
- Add carefully 10ml of Acetyl chloride to the Beaker,it produces the heat instantly,then the crude product is formed.
- Filter the formed product,washed and dried.

SYNTHESIS OF DIBENZAL ACETONE BY GREEN METHOD:

- Take 1ml benzaldehyde in a conical flask and add 30ml of acetone and 15ml of alcohol
- And shake together for 2 mins
- And add 30ml of NAOH solution
- Shake vigorously for 10min
- Along with simultaneously pressure release
- The reaction mixture was cooled in ice
- Pale yellow solid was filtered by filter paper
- Dry the product and weigh it
- Recrystallized from ethanol

SYNTHESIS OF DIBENZALACETONE BY CONVENTIONAL METHOD

- Take benzaldehyde 3.15gm in conical flask and add acetone 2.3ml and add 25ml of ethanol and also add 30ml of distilled water
- Constant stirring the solution with the glass rod
- Take 50ml beaker and pour 3.2ml benzaldehyde and add 2.3 ml of acetone
- Stirring continuously and takeout half of the volume of above solution

- And add it to the NAOH solution in beaker
- With continuous stirring
- Temperature less than 20 is maintained
- Again 15 min stirring after white fluppy precipitate is observed
- Again continuous stirring
- Finally pale precipitate is seen
- Filter the precipitate and get the product

Results and discussion

Comparative studies are done for the green chemical synthesis and conventional synthesis of products acetanilide and Dibenzalacetone. The following are the results obtained:

7.1 Acetanilide:

7.1.1 product obtained by Green Chemical synthesis:

CRUDE DRUG



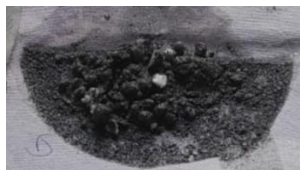
↓

POWDERED DRUG



↓

DECOLOURIZING BY USING CHARCOAL



↓

CRYSTALS OF ACETANILIDE



- The weight of the drug obtained by green chemical synthesis method is found to be : 5.20gms

7.1.2 Product obtained by Conventional synthesis:

CRUDE PRODUCT



↓

POWDERED DRUG



↓

DECOLOURIZING BY USING CHARCOAL





↓





CRYSTALS OF ACETANILIDE



➤ The weight of the drug obtained by conventional method is found to be: 4.18gms

7.1.3 Evaluation parameters :

S.NO	PARAMETER	RESULTS FOR GREEN PRODUCT	RESULTS FOR CONVENTIONAL PRODUCT	Remarks
1.	Assay by UV – Visible Spectroscopy	 <p>Absorbance:280 And its purity was found to be 99%</p>	 <p>Absorbance:242 And its purity was found to be 98%</p>	Purity by both the methods are within the acceptance criteria

2.	Identification test: azodie test	Bright Red color is obtained 	Red color is obtained 	Tests passed
3.	Melting point: Melting point apparatus	113-115°C 	115-117°C 	Within the acceptance criteria and reflects the purity of the drug
4.	DENSITY	1.219g/cm ³	1.259g/cm ³	With in the limits
5.	SOLUBILITY	Soluble in water(<0.56g/100ml)and also soluble in ETHANOL,DIETHYL ETHER,ACETONE,BENZENE	Soluble in water(<0.46g/100ml) and also soluble in ETHANOL,DIETHYL ETHER,ACETONE,BENZENE	Solubility also reflects the purity of the drug, more purity shows more solubility

7.2 Dibenzalacetone:

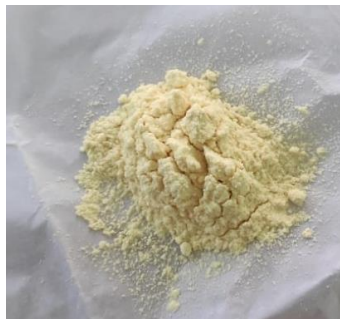
7.2.1 product obtained by Green Chemical synthesis:

PRODUCT OF DIBENZALACETONE



↓

POWDERED DRUG



- The weight of the drug obtained by green chemical synthesis method is found to be : 6.16gms

7.2.2 Product obtained by Conventional synthesis:

PRODUCT OF DIBEZALACETONE



↓







POWDERED DRUG



- The weight of the drug obtained by conventional method is found to be: 5.10gms

7.2.3 Evaluation parameters :

S.NO	PARAMETER	RESULTS FOR GREEN PRODUCT	FOR	GREEN	RESULTS FOR CONVENTIONAL PRODUCT	FOR	Remarks
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1.	Assay by UV – Visible Spectroscopy	 <p>Absorbance:400 And its purity was found to be 101%</p>	 <p>Absorbance:269 And its purity was found to be 99%</p>	Purity by both the methods are within the acceptance criteria
2.	Identification test:	<p>Pure white color is obtained</p> 	<p>yellowish white color is obtained</p> 	Tests passed
3.	Melting point: Melting point apparatus	<p>110-111°C</p> 	<p>112-115°C</p> 	Within the acceptance criteria and reflects the purity of the drug
4.	DENSITY	<p>1.101g/cm²</p>	<p>1.120g/cm³</p>	With in the limits
5.	SOLUBILITY	<p>InSoluble in water</p>	<p>Soluble in ETHANOL</p>	Solubility also reflects the purity of the drug, more purity shows more solubility.

Limitations:

- 1) Green chemical synthesis implementing costs are very high
- 2) Lack of information about green chemicals and green chemical synthesis procedures for more drugs
- 3) No known alternative green chemicals or raw materials to input in green chemicals synthesis
- 4) No known alternative procedures or process technology about green chemical synthesis

6. CONCLUSION:

Green chemistry is mainly focus on the synthesis of chemical compounds to get maximum yield with minimal waste, atom economy, usage of less toxic chemicals, less wastage, high efficiency, green catalysts. The basic aim of our study is to perform comparative studies of green chemical synthesis and conventional synthesis of *Acetanilide* and *Dibenzalacetone* and its evaluation parameters. Green chemical synthesis is a green tool for organic synthesis providing clean, economic enhanced rate of reaction and percentage of yield. In present study *Acetanilide* and *Dibenzalacetone* was taken to performing the studies of green chemical synthesis and conventional synthesis methods. Green chemical synthesis method was found to be highly efficient, Where all the compounds are synthesized and characterized by using UV-spectroscopy and melting point. By these comparative studies we found that acetanilide and dibenzalacetone was prepared by green chemical synthesis shows more efficiency, less reaction time, less usage of amount of solvents and high yields, more purity, more activity, and less wastage, less hazardous when compared with the conventional synthesis methods. Acetanilide is synthetic organic compound introduced in the therapy in 1886 as fever reducing agent, its helps in relieving pain. And used as an alternative to aspirin for treating headache, menstrual cramps, and rheumatism. Dibenzalacetone have anti oxidant activity, a key component in sunscreens and ligand in organo metallic chemistry.

Finally we conclude that green chemical synthesis is an eco-friendly method and suggested to adapt these methods furthermore in future to reduce the pollution of the environment.

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