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Green Synthesis and Characterization of Schiff Base by Using Natural Acid Catalysts

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Abstract: *The utilization of green chemistry techniques dramatically reduces chemical wastes and reaction time as recently have been proven in several organic synthesis and chemical transformations. To illustrate these advantages in the synthesis of organic heterocycles, various environmentally benign protocols that involve greener alternatives have been studied. The objective of present research work has also used green methodologies for synthesis of Schiff bases. Conventionally synthesis of Schiff base is carried out with or without acid catalyst and sometimes by refluxing the mixture of aldehyde (or ketone) and amine in organic medium. Present synthesis involves the use of fruit juice of Citrus limetta, Vitis lanata and aqueous extract of Mangifera indica as natural acid catalysts.*
The synthesized product was identified by its physical properties, melting point, TLC and characterized by IR spectrometry and 1H NMR spectroscopy. Compared with traditional methods, these methods were more convenient and provided higher yield (82-93%), shows maximum efficiency, held without generation of pollution in shorter reaction time, safer to analyst, low cost and simple to run..

Keywords: Green chemistry, Schiff bases, natural acid catalysts, IR spectrometry, ^{1}H NMR spectroscopy

1. Introduction

Green chemistry is the branch of chemistry that involves tools techniques and technologies. It is helpful to chemists and chemical engineers in research, development and production, for development of more eco-friendly and efficient products which may also have significant financial benefits. It is going to now become an essential tool in the synthetic chemistry. It is a new way of looking at organic synthesis and the design of drug molecules, offering important environmental and economic advantages over traditional synthetic processes.

The recent interest in green chemistry has posed a new challenge for organic synthesis in that new reaction conditions need to be found which reduce the emission of volatile organic solvents and the use of hazardous toxic chemicals. They improve selectivity, reduces reaction time, and simplifies separation and purification of products than the conventional methods. Organic synthetic procedures use organic solvents like benzene and chlorinated hydrocarbons, which have created havoc to the environment because of their toxic and volatile nature. Solvent free reactions usually need shorter reaction times, simpler reactors, resulting simpler and more efficient work up procedures, more improved selectivities and easier separations and purifications than conventional solvents. The role of naturally available fruit juice in organic synthesis has attracted the interest of chemists, particularly from the view of green chemistry. This shows versatile synthetic applications of fruit juice as a biocatalyst in chemical transformation.

Nowadays the solvent-free approach to the synthesis of molecules becomes an attractive one since the majority of solvents are either toxic or flammable and adds considerably to the cost of overall synthesis. In many cases, the solvent-free approach improves selectivity, reduces reaction time, and simplifies separation and purification of products than the conventional methods. Natural acid is non-polluting and does not employ any lethal materials, quantifying if as a green approach for the synthesis of Schiff bases.

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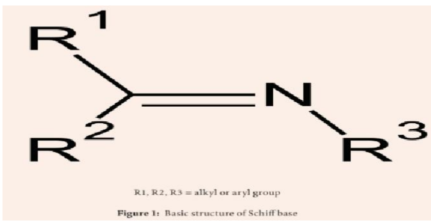
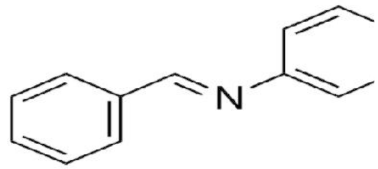
2. LITERATURE REVIEW

Schiff bases, known as Imines are compounds containing azomethine group -(HC=N)- and represented by the general formula $R_3R_2C{=}NR_1$. They are the condensed products of aldehydes or ketones and were first reported by Hugo Schiff in 1864. Originally, the classical synthetic route for synthesis of Schiff bases was reported by Schiff which involves condensation of primary amines with carbonyl compounds under azeotropic distillation with the simultaneous removal of water.





N- benzylidene aniline

Interests in these compounds are largely due to their structural similarities with natural biological substances and relatively simple procedures of synthesis as well as synthetic flexibility that enable the design of suitable structural properties. They are well known intermediate for the preparation of azetidinone, thiazolidinone, formazone, arylacetamide, metal complexes and many other derivatives. The Schiff bases constitute one of the most active classes of the compounds possessing diversified biological applications such as antitubercular, anticancer, antibacterial, anti-inflammatory, antifungal, antitumor, diuretic, insecticidal, herbicidal, anthelmintic, anti-HIV, antiproliferative, anticonvulsant, antihypertensive and antiparasitic activities.

The Schiff's base derivatives have been extensively investigated for more than a century and employed in different aspects including magneto chemistry, non-linear optics, photo physical studies, catalysis, materials chemistry, chemical analysis, absorption and transport of oxygen. Due to these beneficial properties, concern for the environmental demands and strong interest in the development of green chemistry, new sustainable catalysts and new environmentally benign processes have been investigated which are both economically and technologically feasible. Present study also involves some eco-friendly and inexpensive natural catalysts like **grapes (*Vitis lanata*) juice, sweet lemon (*Citrus limetta*) juice and aqueous extract of mango (*Mangifera indica*)** for the synthesis of Schiff bases.

3. METHODOLOGY

Preparation of catalyst: -

Grapes, sweet lime and unripe mango fruits were purchased locally, then grapes were pressed into fruit juicer and filtered with cotton to get liquid juice.

While sweet lime fruits were peeled off with knife and fruit slices were pressed into fruit juicer to get semisolid mass which was then filtered with cotton to get liquid juice to used as catalyst.

The upper shell of unripe Mango fruit was removed and the hard fleshy green material (5 g) was boiled with water (100 ml), cooled and filtered with muslin cloth to get clear liquid portion to used as catalyst for the synthesis.

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Grapes (*Vitis lanata*) juice, sweet lemon (*Citrus limetta*) juice and aqueous extract of Mango (*Mangifera indica*) prepared and stored

Synthesis of Schiff bases with grapes juice, sweet lemon juice and aq. extract of unripe mango under solvent free condition by stirring method: -

The equimolar amount of benzaldehyde (0.04 mol, 4.06 ml) with aniline (0.04 mol, 3.65 ml) was taken in different beakers.

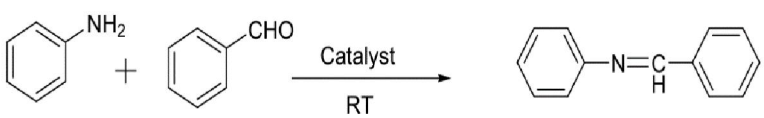
In those reaction mixtures natural acid catalyst i.e. grapes juice was added in variable amounts (1.0 ml, 2.0 ml, 3.0 ml) and then kept for 5-10 minutes.

Further each reaction mixture was stirred for 2 - 4 minutes at room temperature pale yellow solid crude product was appeared after completion of reaction.

This crude product was washed with distilled water and purified by recrystallization with minimum amount of ethanol.

The same procedure is repeated with sweet lemon juice and aqueous extract of mango.

Melting point of the products were measured by open capillary method which was further identified and purified with the help of TLC and confirmed by using IR spectra and ^{1}H NMR spectra.



Scheme 1. Schiff base reaction between benzaldehyde and aniline.

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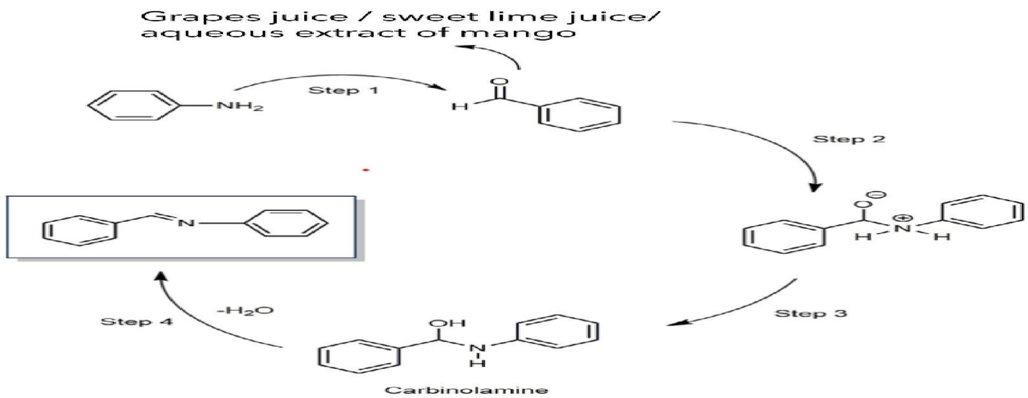
Mechanism



Mechanism for Schiff base N-Benzylidene aniline synthesis in the presence of acid catalysts

4. RESULT AND DISCUSSION

Calculations: -

A) Theoretical Yield :-

93.13 gm of aniline = 181.24 gm of N-benzylidene aniline

Therefore, 3.65 ml of aniline = (3.65 * 181.24) / 93.13 = 7.10 gm of N-benzylidene aniline

B) Practical Yield :-

i) Grape juice

% Weight of crude product = **93.23 %**

ii) Sweet lemon juice

% Weight of crude product = **88.45 %**

iii) Aqueous extract of unripe mango

% Weight of crude product = **91.51 %**

Yields of the N-benzylidene aniline obtained with different amount of natural acid catalysts

Title/ Sr. No.	Amount of catalyst (ml)	Product yield obtained with grapes juice		Product yield obtained with sweet lemon juice		Product yield obtained with aqueous extract of unripe mango	
		Product Yield (gm)	Percentage Yield	Product Yield (gm)	Percentage Yield	Product Yield (gm)	Percentage Yield
1	1.0	6.62	93.23	6.28	88.45	6.51	91.69
2	2.0	6.35	89.43	6.08	85.63	6.27	88.30
3	3.0	6.10	85.91	5.85	82.39	5.88	82.81

Physical characteristics of Product

Name and Chemical formula	N- benzylidene aniline ($C_{13}H_{11}N$)
Physical state	Crystalline Solid
Colour	Pale yellow
Smell	Disagreeable

Theoretical Yield	7.10 gm
Percentage Yield	93.23 %
Solubility	Insoluble in distilled water and soluble in methanol, ethanol and chloroform
Molecular Weight	181.24 g/mol
Melting Point	54^{o} C
Rf value	0.64

Spectral Discussion

FT-IR spectra of compound was Scanned on Bruker Spectrometer and ^{1}H NMR spectra was recorded in $CDCl_3$ on AVANCE- 400 MHz instrument using TMS as an internal standard.

FT- IR

IR spectra of all Schiff bases show characteristic band in the region 1640-1690 cm^{-1} due to -C=N stretching vibration.
And band around 3000-3100 cm^{-1} due to aromatic alkene -C=C- stretching.
Stretching around 700 cm^{-1} and 750 cm^{-1} shows two mono-substitutions of benzene rings.

^{1}H NMR

^{1}H NMR spectra of Schiff base shows singlet of azomethine group at δ 8.5 ppm (s, 1H).
And aromatic protons in benzene rings observed at 8.0 ppm (m, 2 H), 7.5 ppm (m, 3 H), 7.4 ppm (m, 2 H), 7.3 ppm (m, 3 H).

5. CONCLUSION

The present work focuses the importance of fruit juice as natural and biocatalyst in organic transformations.
The growing interest of fruit juice in organic synthesis is mainly due to their acidic properties, enzymatic activity, benign environmental character, inexpensive, and commercial availability.
The catalytic activity including the application of fruit juices in various organic transformations such as formation of C=C, C-N bonds in different synthetically important organic compounds have been studied.
The IR band at 1640 cm^{-1} is due to the presence of azomethine group, this confirms the formation of Schiff base.
This natural acid catalyst technique is non-polluting and does not employ any toxic materials, quantifying it as a green approach for the synthesis of Schiff base.
In addition to this, compared to traditional methods, this new method is clean, safe and more eco-friendly, involving mild reaction conditions and simple workup.
The reaction conditions such as reaction time, use of hazardous solvents can be reduced by maintaining good yield of product.

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