

# MICROWAVE ASSISTED GREEN SYNTHESIS OF SUCCINIC ACID DERIVATIVES FROM FURFURAL AND STUDY OF THEIR ANTIBACTERIAL ACTIVITY

Ganesh N Yallappa  
Dept. of Chemistry  
Jain Institute of Technology  
Davangere-577003  
Karnataka, India  
gindi.ny1988@gmail.com

Ganesh D B  
Dept. of Mechanical Engineering  
Jain Institute of Technology  
Davangere-577003  
Karnataka, India

Nataraja G  
Dept. of Chemistry  
Jain Institute of Technology  
Davangere-577003  
Karnataka, India

Niranjana M  
Dept. of Physics  
Jain Institute of Technology  
Davangere-577003  
Karnataka, India

Tanuja S B  
Dept. of Chemistry  
Jain Institute of Technology  
Davangere-577003  
Karnataka, India

Prakash M Walvalkar  
Dept. of MBA  
Jain Institute of Technology  
Davangere-577003  
Karnataka, India

Spoorthi S  
Dept. of Civil Engineering  
Jain Institute of Technology  
Davangere-577003

## Abstract

Green oxidation of inedible biomass based furfuraldehyde [furfural] to succinic acid [SA] using hydrogen peroxide as an oxidizing agent called as green oxidant takes place as following.

Ring opening reaction happens when furfural is added with hydrogen peroxide [H<sub>2</sub>O<sub>2</sub>], under certain temperature the ring opening of furfural leads to the formation of succinic acid as a product. The completion of the reaction can be checked through thin layer chromatography. We studied antibacterial activity for synthesized Succinic acid, 2-iodo succinic acid.

**Key words-**Succinic acid derivatives, Green synthesis, Microwave synthesis, Hydrogen peroxide, furfural.

## I. INTRODUCTION

Green chemistry has been playing very great role in the field of organic chemistry for the synthesis of organic compounds. We planned for the green synthesis of Succinic acid by a simple microwave method [1-2]. Succinic acid was prepared by the starting material Furfural which undergoes oxidation and Iodination [3-5]. The reaction was done by microwave irradiation. Microwave energy provided the energy for chemical reaction and reaction time was very less compared to conventional method. Furfural is a five membered aldehyde cyclic ring which upon treated with H<sub>2</sub>O<sub>2</sub> undergoes oxidation and ring opening takes place to form succinic acid. Later the same was treated with Potassium Iodide to undergo Iodination to form Iodo- succinic acid [6-7].

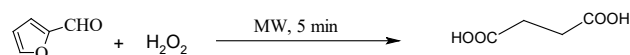
## II. MATERIALS AND METHODS

## EXPERIMENTAL WORK

### General method for the Synthesis of Succinic acid and its substituents Synthesis of Succinic acid:

Firstly, succinic acid has been prepared by taking the reactant as furfural, which is an organic compound. 9ml of Furfural is measured and taken into a clean beaker and to this 25 ml of hydrogen peroxide has been added, which acts as an oxidizing agent and the reaction mixture of furfural and H<sub>2</sub>O<sub>2</sub> is heated in the microwave oven for sometimes until the reaction completes and the completion of the reaction is checked through TLC. The obtained succinic acid product is filtered and washed with ethanol and dried, then weighed and its melting point is observed and the compound is collected in a sample bottle [8-11].

### Reaction Scheme-1

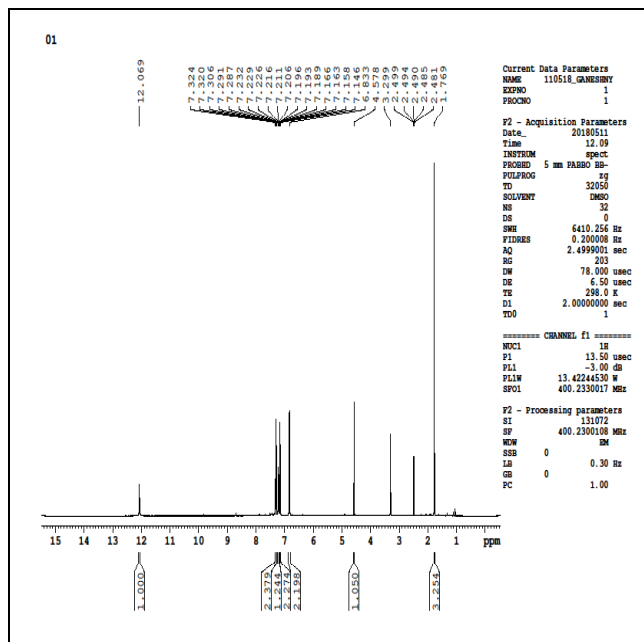
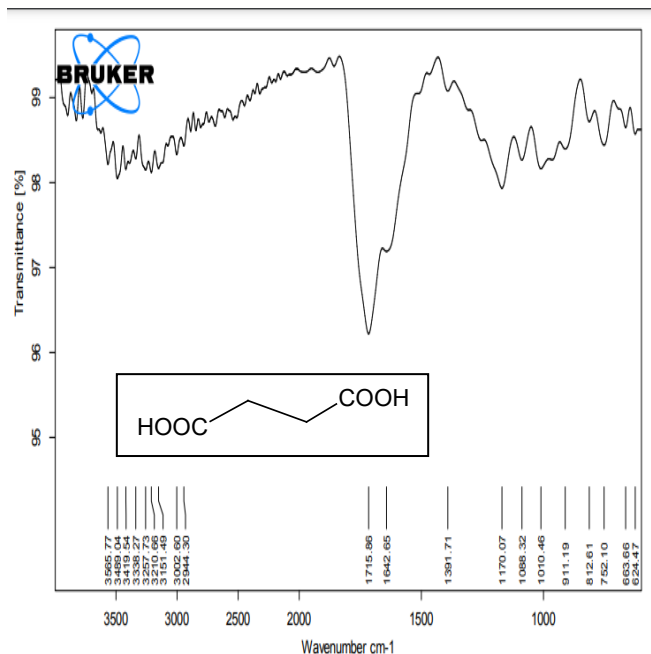
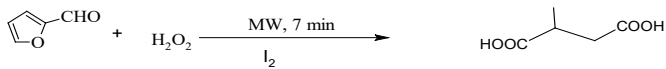


### Synthesis of 2-Iodo Succinic acid:

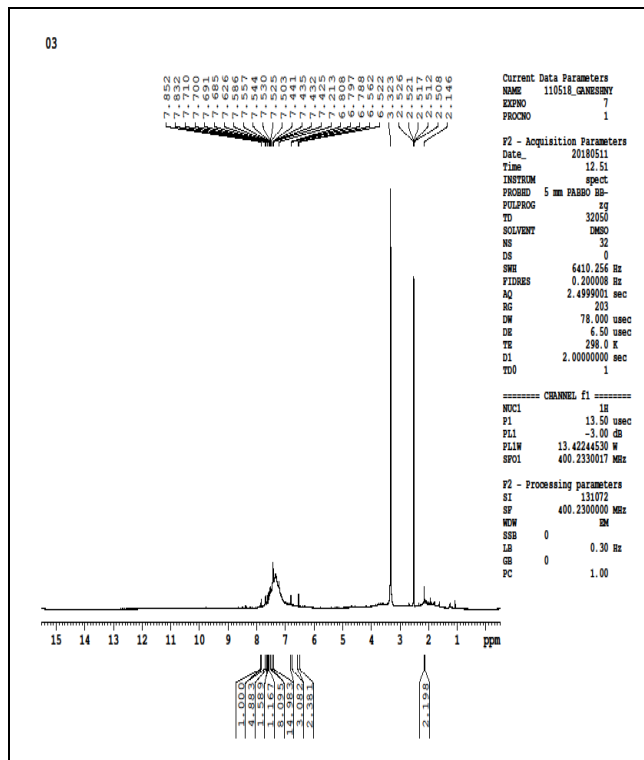
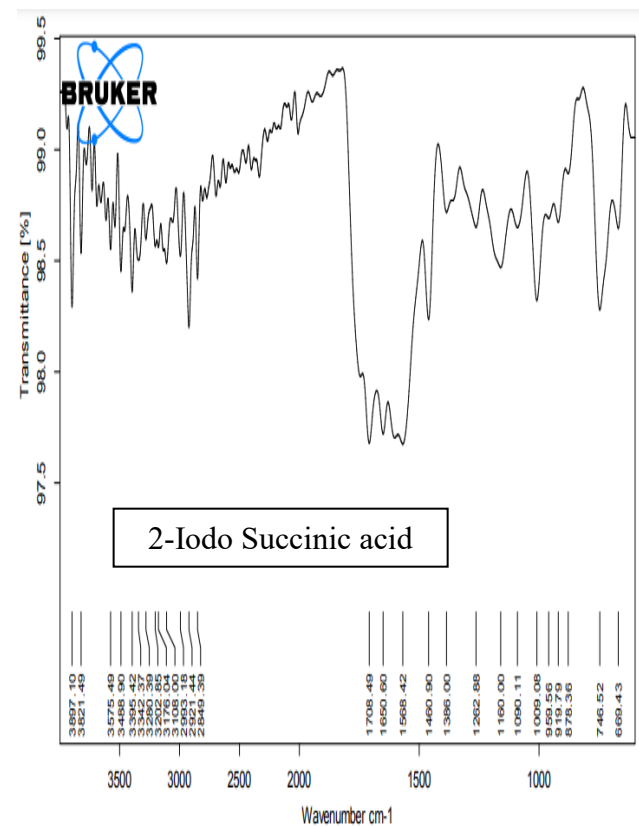
0.166g of potassium iodide is dissolved in 10ml of ethanol and the solution is added to the 9ml of furfural in a beaker the reaction mixture is heated for some time until the reaction completes. The completion of the reaction is checked through TLC. The obtained Iodo furfural product is filtered and washed with ethanol and dried, then weighed and its melting

point is observed and the compound is collected in a sample bottle<sup>10</sup>.

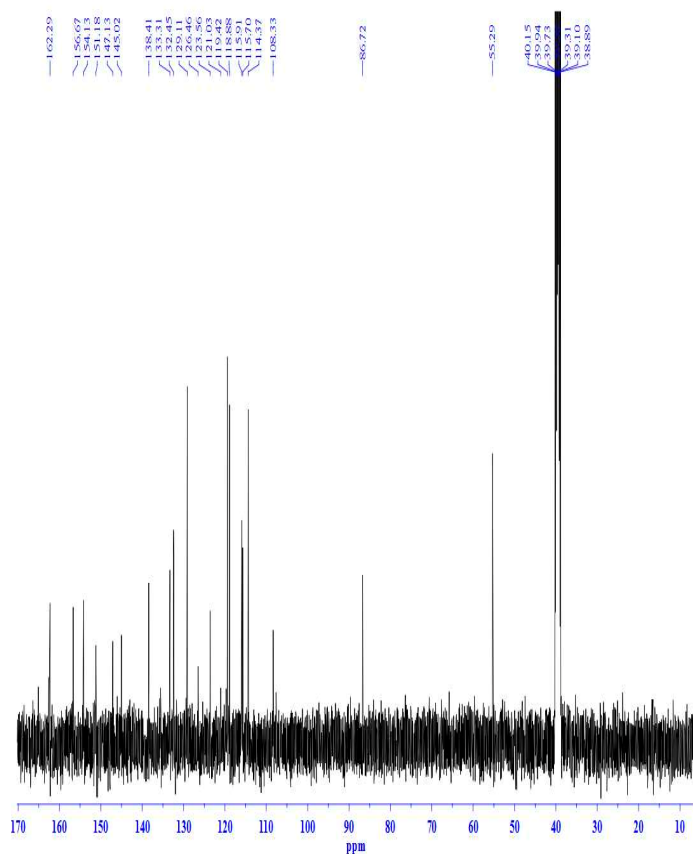
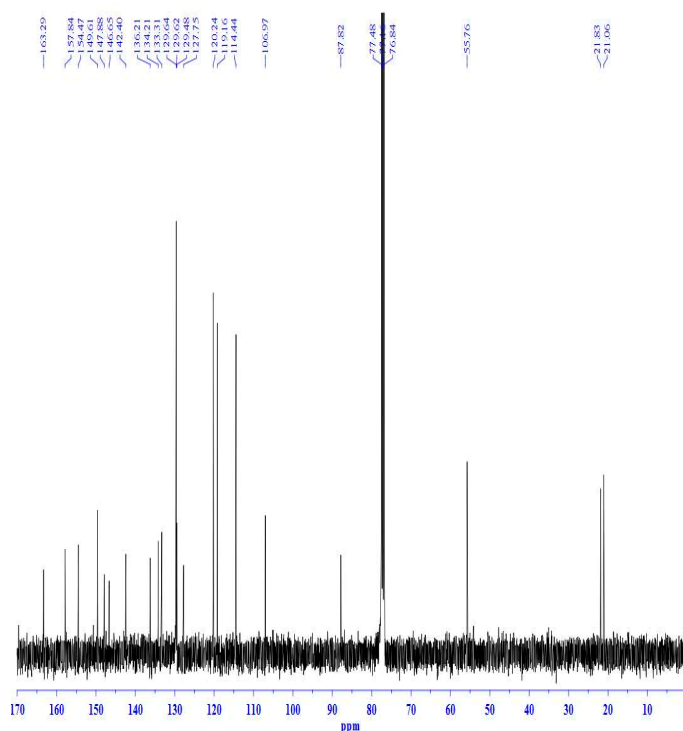
Reaction Scheme-2:



<sup>1</sup>H NMR of Succinic acid



<sup>1</sup>H NMR of 2-iodo-Succinic acid

 $^{13}\text{C-NMR}$  of Succinic acid $^{13}\text{C-NMR}$  of 2-iodo-Succinic acid

### III. RESULTS AND DISCUSSION

**Table 1. Reaction time of Succinic acid and its substituents**

Compounds	Reaction Time in Minutes (MW)	Yield (%)	Color	m.p.
Succinic acid	5	88.00	Brown	181°C
2-iodo Succinic acid	7	74.00	Brown	198°C

**Succinic acid:** Brown solid, Yield= 88.00%, m.p: 181°C, IR (KBr)  $\text{Cm}^{-1}$ : 3395 (m, Carboxylic acids, stretch), 1715 (S, Carboxylic acids), 3002 (C-H, stretch), 1391.71 (m,  $\text{CH}_2$ ).

**2-Iodo Succinic acid:** Brown solid, Yield= 74%, m.p: 198°C, IR (KBr)  $\text{Cm}^{-1}$ : 3342 (m, Carboxylic acids, stretch), 1708 (S, Carboxylic acids), 2993 (C-H, stretch), 1460.71 (m,  $\text{CH}_2$ ).

In the table-1, Succinic acid formed in 5 mins under microwave irradiation and having 88.00% yield.

Similarly, 2-iodo succinic acid completed its reaction in 7 mins and having yield 74.00%.

Succinic acid possesses various applications in the field of medicine. After literature studies, Succinic acid prepared to use for the biological activities. Hence, we planned to study antibacterial activity [12-13].

#### Biological activity

##### Procedure

##### Well diffusion method

In Fig 1, the mechanism has been studied that how the compounds inhibit the bacteria. Prepare agar medium by taking proportions of calculated weight of Peptone, Beef extract, NaOH Solution in conical flask.

Sterilize the petri plates, pour the agar medium and dry. Keep these petri plates in incubation. Make the desired size of wells to the dried agar medium. Scrub the bacterial cultures on the plates. Keep the plates for incubation to grow the bacteria.

Apply the synthesized compounds Succinic acid and 2-iodo-succinic acid of desired concentrations on the wells. Keep them for 24hrs incubation and measure the zone of inhibition of compounds which inhibit the bacterial growth.

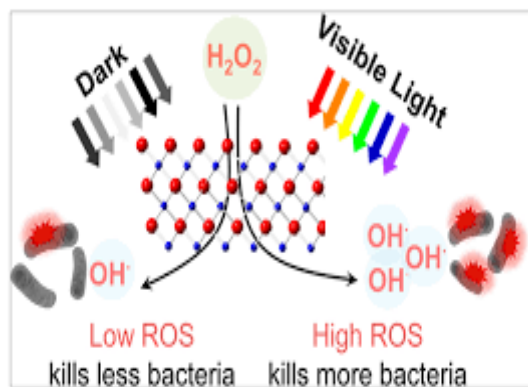


Fig 1. Mechanism of Bacterial inhibition

Firstly, desired bacterial cultures for the study required. After the studies on bacteria E.Coli, Streptococcus, Staphylo cocci, afforded good positive results. In fig 2, zone of inhibition has been marked in the petri plates. In fig 3, MTT image of inhibition for succinic acid has been displayed [14-15].

In the table 2, Succinic acid showed zone of inhibition 16mm for E.Coli, 24mm for Staphylococcus aureus and 17mm for Bacillus cereus. Similarly, 2-iodo succinic acid exhibited 27mm for E.Coli, 14mm for Staphylococcus aureus and 18mm for Bacillus cereus.

**Table2. Zone of Inhibition of compounds Succinic Acid and 2-Iodo-Succinic acid**

Compound Name	Zone of Inhibition (mm)		
	<i>E.Coli</i>	<i>Staphylococcus aureus</i>	<i>Bacillus cereus</i>
Succinic Acid	16	24	17
2-Iodo-Succinic Acid	27	14	18

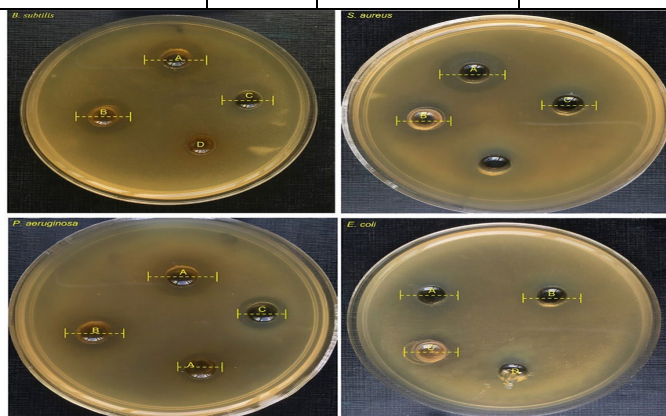


Fig 2. Images of Inhibition in agar media



Fig 3. MTT assay

#### IV. CONCLUSION

- We followed Green chemistry for the synthesis of succinic acid and its substituents so that this work is mainly on the concern of environment protection.
- The FT-IR characterization confirmed the structure of all synthesized compounds.

#### V. ACKNOWLEDGEMENT

All authors express sincere gratitude to Jain Institute of Technology, Davangere-577003 for the support of the work.

#### REFERENCES

- [1]. P. Penczek, P. Czub, J. Pielichowski. "Unsaturated polyester resins: Chemistry and technology". Adv. Polym. Sci., vol. 184, pp 1–95, 2005.
- [2]. F. R. Jones. "Unsaturated Polyester Resins. In Brydson's Plastics Materials", 8th ed.; Elsevier: Amsterdam, The Netherlands, pp. 743–772. ISBN 9780323358248.
- [3]. M. Malik, V. Choudhary, I.K.Varma. "Current Status of Unsaturated Polyester Resins". J. Macromol. Sci. Part C Polym. Rev., vol. 40, pp 139–165, 2000.
- [4]. Q. Li, S. Ma, X. Xu, J. Zhu. "Bio-based Unsaturated Polyesters. In Unsaturated Polyester Resins", Elsevier: Amsterdam, The Netherlands, pp. 515–555. ISBN 9780128161296.
- [5]. Y. Zhu, C. Romain, C. K. Williams. "Sustainable polymers from renewable resources". Nature. vol. 540, pp 354–362, 2016.
- [6]. N. Ganesh Yallappa, S. Rajendra Prasad, G. Nataraja, G. Megha, S.V. Dhanyashree, P. Leela, G T Manukumar. Microwave assisted Green synthesis of Paracetamol, Aspirin and Study of their Pharmacokinetics". International Journal of

- Engineering Research and Technology, vol. 10 (11), pp 494-496, 2022.
- [7]. N. Ganesh Yallappa, D. Nagaraja, U. Chandrashekhar. "Nanocatalyzed Green synthesis of Pyrazole derivatives & its biological activity as EAC receptor antagonists". Pharmacophore, vol. 28(3), pp 28-32, 2019.
- [8]. N. Ganesh Yallappa, S. Rajendra Prasad, Gayatri Vaidya. "Design and synthesis of pyrazole derivatives for *in vitro* screening to protect angiotensin-converting enzyme 2 human cells against COVID-19". Curr. Medicine research and practice, vol. 11(5), pp 206-210, 2021.
- [9] Mudradi Sudheer, Ravikantha Prabhu, Kandavalli Raju, and Thirumaleshwara Bhat, "Effect of Filler Content on the Performance of Epoxy/PTW Composites" Advances in Materials Science and Engineering, Volume 2014, pp.1-11.
- [10] J. S. Sidhu, G. S. Lathkar, and S. B. Sharma, "Mechanical properties of micro tungsten disulphide particles filled epoxy composite and its resistance against sliding wear" Malaysian Polymer Journal, Vol. 9 No. 1, pp. 24-32, 2014.
- [11] L. Bharath, M.S. Reddy, H.N. Girisha, G. Balakumar, "Influence of process parameters on tensile strength and hardness of AW2024/B4C composite using Taguchi's technique" Materials Today: Proceedings, Elsevier ISSN:2214-7853.
- [12] S.R. Ranganatha, Dr. V. S. Ramamurthy, "Investigation on mechanical behavior of filler Al<sub>2</sub>O<sub>3</sub> in CFRP composites" IJAET, E-ISSN 0976-3945, PP.105-107.
- [13] Yibowei Moses, Ichetaonye Simon, Idehenre Maxwell, "Mechanical Properties of Carbon Fibre and Metal Particles Filled Epoxy Composite" IJETAE, ISSN:2250-2459, Vol.3, Issue 11, pp.664-667.
- [14]. M. Segun. Adedayo and A. Modupe Onitiri, "Tensile Properties of Iron Ore Tailings Filled Epoxy Composites" The West Indian Journal of Engineering Vol.35, No.1, pp.51-59, July 2012.
- [15] J. Stabik, A. Dybowska, M. Chomiak, "Polymer composites filled with powders as polymer graded materials" JAMME, Volume 43, Issue 1, pp.153-161, November 2010.