IJARSCT
International Journal of Advanced Research in Science, Communication and Technology
International Open-Access, Double-Blind, Peer-Reviewed, Refereed, Multidisciplinary Online Journal
ISSN: 2581-9429
Volume 5, Issue 11, May 2025
Impact Factor: 7.67

Synthesis, Characteristics, And Pharmacology Activity of Newly Synthesized 3-(4-Methylphenyl) Prop-2-Enoic Acid by using Substitution from P-Tolualdehyde

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Abstract: *The p-methoxycinnamic acid (p-MCA) is one of the most studied phenylpropanoids with high Importance not only in the wide spectrum of therapeutic activities but also its potential application for the. Compound derived from plants exhibits a wide range food industry. This natural of biologically useful properties; therefore, during the last two decades it has been extensively tested for therapeutic and nutraceutical applications This study aimed to synthesize p-methoxy cinnamic acid through the Perkin reaction and to determine its activity as a photo protective and antibacterial agent against The PMCA compound was synthesized by reacting p-methoxy benzaldehyde with acetic anhydride using a sodium acetate catalyst in a sonicator at 500C for 60 minutes. The synthesized was a white precipitate with a % yield of 2.09% and a melting point of 172-1750C. ATR-FTIR identified this compound with several functional groups, C-O, OH carboxylic acid, para-substituted benzene, and C-C. Analysis by GC-MS showed a single peak at a retention time of 11.710 minutes with m/z 178. Characterization of this compound by 1H-NMR spectrometry showed several chemical shifts showing the presence of OH groups of carboxylic acids, C-C groups, aromatic benzene groups, and methoxy. The results of this characterization indicated that the synthesis product..*

Keywords: P-methoxycinnamic acid, antibacterial activity, carboxylic acid, Solid state NMR, FT-IR

I. INTRODUCTION

p-Methoxy cinnamic acid is a derivative of cinnamic acid, where a methoxy group is attached at the para position. This compound is known to have various biological activities, including antibacterial, anti-inflammatory, antic-cancer antioxidant, antidiabetic, hepatoprotective, Neuroprotective, and chemo preventive effects. P-Methoxy cinnamic acid can be produced by hydrolyzing ethyl p-methoxycinnamate, which is isolated from natural sources such as aromatic ginger or through the Perkin reaction. The Perkin reaction is one of the most commonly used methods for synthesizing cinnamic acid derivatives.

The Perkin reaction offers the advantage of a simple process and easily accessible starting materials. In the conventional method, p-methoxy cinnamic acid is synthesized by reacting p-methoxy benzaldehyde (an aldehyde) with acetic anhydride, using anhydrous sodium acetate as a catalyst. However, this process typically requires high temperatures, long reaction times, and often results in low yields Therefore, alternative approaches, such as the use of ultrasonic waves, are being explored.

Ultrasonic waves create a phenomenon called cavitation the formation, growth, and collapse of bubbles which can generate extremely high temperatures (around 5000 K) and pressures (up to 1000 atm), leading to the breaking of chemical bonds. In recent years, so no chemical methods have gained popularity because they offer better control

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compared to traditional methods Acid derivatives possess antibacterial activity against E. coli and can also act as sunscreens. Therefore, it is expected that p-methoxy cinnamic acid may show even better biological activities.

This research aims to synthesize p-methoxy cinnamic acid using a Perkin reaction assisted by ultrasonic waves and to evaluate its antioxidant, sunscreen, and antifungal activities against. The goal is to develop synthetic medicinal raw materials that can be used as photoprotective and antifungal agents.

DRUG PROFILE:

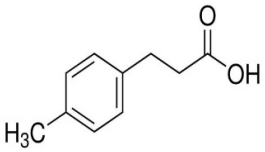
Drug name	P methlycinnamic acid
IUPAC name	3-(4-Methylphenyl)prop-2-enoic acid
Molecular formula	C10H10O2
Molecular weight	162.19 g/mol
Structure	
Theoretical yield	6.75g
Practical yield	6.2 g
Appearance	White to off-white crystalline solid
Solubility	In water and ethanol
Category	Antimicrobial, Anti inflammatory

Table: 1.1 Drug profile

MATERIALS AND METHOD

Chemicals	Apparatus	Equipment
Toluene	Round bottom flask	Water bath
Fecl3	Magnetic stirrer	Weighing machine
Water	Reflux condenser	Thermometer
H2so4	Beaker	Hot air oven
Sodium hydroxide	Measuring cylinder	FT -IR
Sodium acetate	Pipette	Computer
Conc . hydrochloride acid	Petri dish	
Ethanol	Funnel	
Benzaldehyde	Filter paper	
Acetone	Tripod stand	

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Method:

Starting material availability p-Tolualdehyde should be readily available and affordable. Based on the selection criteria, the Perkin reaction was selected as the method for synthesizing p-methylcinnamic acid derivatives. This method involves the reaction of p-tolualdehyde with acetic anhydride and sodium acetate to produce p-methylcinnamic acid.

Reactions:

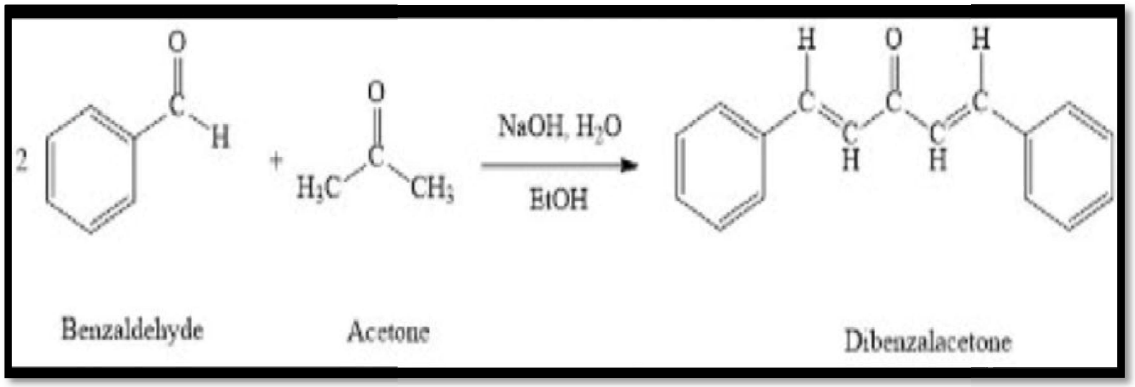

Fig: Reaction 1.1 Synthesis of Benzaldehyde

Procedure:

1. Prepare NaOH Solution:
Dissolve 2–3 g of NaOH pellets in 10 mL of distilled water. Cool the solution to room temperature.

2. Prepare Reaction Mixture:
Mix 10 mL of ethanol with 5 mL of benzaldehyde in a beaker. Add 2.5 mL of acetone to the above mixture.

3. Add Base Catalyst:
Slowly add the cold NaOH solution dropwise to the reaction mixture with constant stirring.

4. Stir and React:
Continue stirring the mixture for 30–60 minutes at room temperature. A yellow precipitate (dibenzalacetone) will start forming.

5. Ice Bath Cooling:
Place the mixture in an ice bath for 10–15 minutes to complete precipitation.

6. Filter the Product:
Filter the solid dibenzalacetone using a Buchner funnel or simple filtration.

7. Wash the Precipitate:
Wash the solid with cold distilled water to remove excess NaOH and impurities.

8. Recrystallization:
Recrystallize the crude product from hot ethanol to purify.

9. Dry the Product:
Dry the pure yellow crystals in an oven or desiccator.

DOI: 10.48175/568

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Procedure Synthesis of Substituted P Tolualdehyde

A) Aldol Condensation (B-hydroxy ketone Formation)

- p-Tolualdehyde (1.0 mol) and
- acetone (2.0 mol) were stirred with
- NaOH (10% aqueous) at room
- Temperature for 2 hours. The product
- Was extracted with ethyl acetate and
- Purified via column chromatography.

B) Hydrazone Formation (C=0 Substitution)

- P-Tolualdehyde was reacted with
- Phenyl hydrazine (1.0 mol) in ethanol with catalytic acetic acid.
- The mixture was refluxed for 2 hours to yield the hydrazone derivative.

C) Grignard Reaction (Alcohol formation)

- P-Tolualdehyde (1.0 mol) was
- Reacted with phenyl magnesium
- Bromide (1.2 mol) in dry ether under inert atmosphere. A stirring3hr at room temperature, the
- Reaction was quenched with
- Ammonium chloride solution and the benzylic alcohol was isolated.

Reaction:

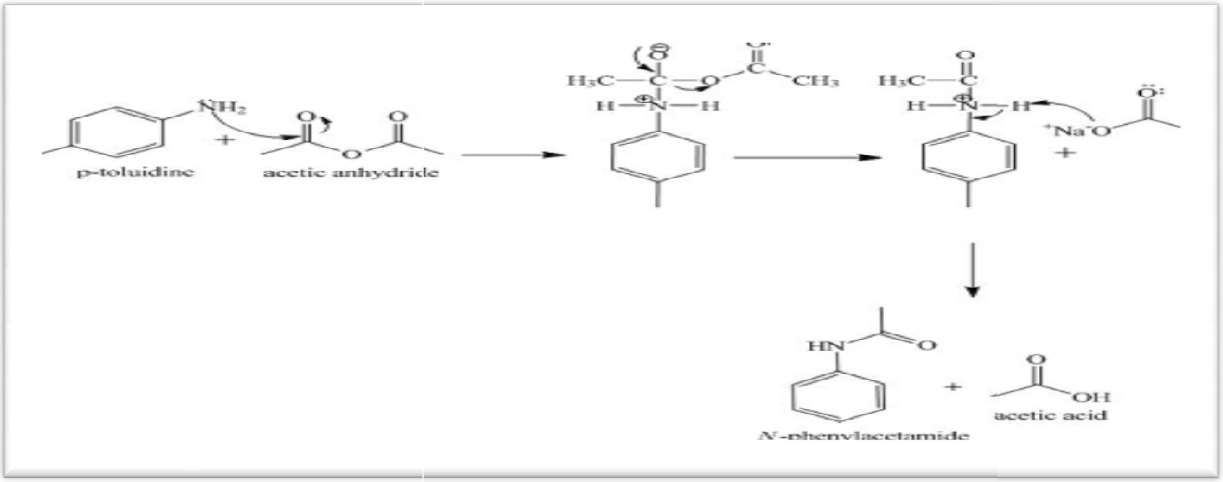

Fig:1.2 Substituent of p Tolualdehyd

Newly synthesized of P Tolualdehyde and P methlycinnamic acid derivatives:

1. Take a 2gm of Tolualdehyde
2. Add Acetic anhydride for 100 ml round bottom flask
3. Add 1ml sodium hydroxide + 2ml sodium acetate, get stirrer the mixture at room temperature for 30 min
4. Mixture get reflux for 4 hrs
5. Cool mixture at room temperature in 10 min
6. Add 2ml of conc Hydrochloride acid get strting mixture in 10 min
7. Separate ethanol layer, in petri dish add 2ml ethanol and dry at product 2 days
8. After 2 days' observation in that obtained yellowish white off colour crystal obtained

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Reaction:

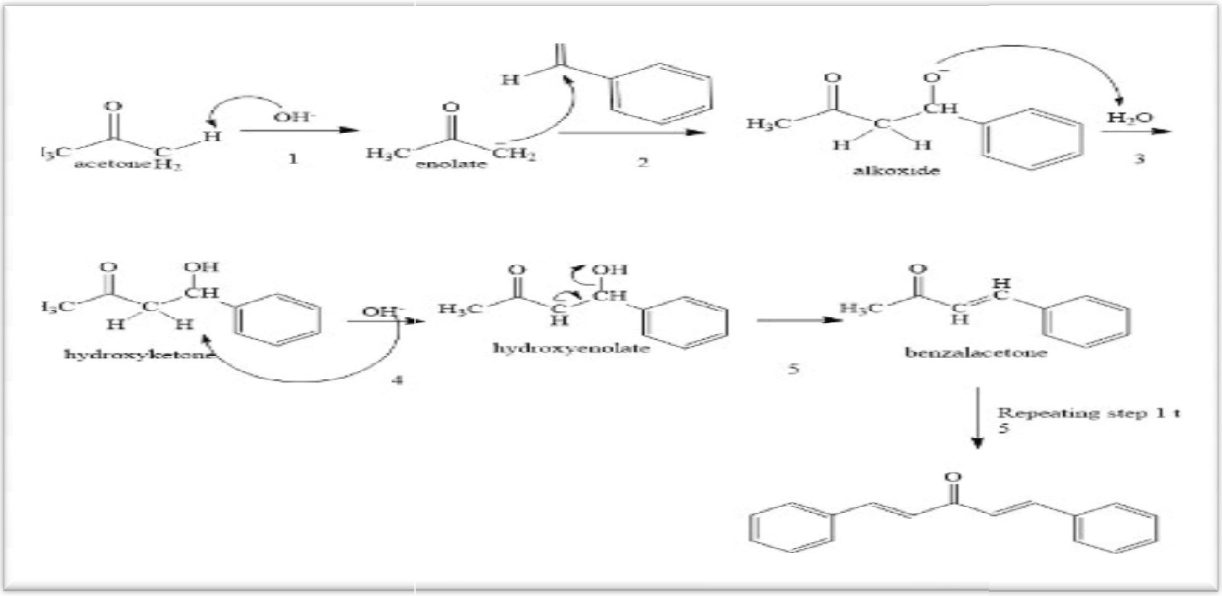

Fig:1.3 Reaction of Hydrazone Formation (C=0 Substitution) and Alcohol formation)

Compound	IUPAC Name	Molecular weight	Melting point
Compound A	P -Tolualdehyde	120.15 g/mol	43-45°C
Compound B	P- methlycinnamic acid	62.19 g/mol	175°C

Table: 1.3 Synthesized derivative

SWISS ADME:

Compound Name	Molecular weight	H-Bonding acceptor	H- Bond Donors	Log p	Violations of Lipinski rule
P methlycinnamic acid	62.19 g/mol	1	2	2.5	0 Violation
P Tolualdehyde	120.15 g/mol	1	0	2.34	0 Violation

Table: 1.4 Swiss ADME

CHARACTERIZATION:

FTIR

Fourier Transform Infrared Spectroscopy (FTIR) is a widely used analytical technique That provides valuable information about the chemical composition and structure Materials. It is a non-destructive technique that works by measuring the interaction between a sample and infrared radiations

The FTIR spectrophotometric analysis, as presented in on firms the presence of functional groups consistent with the target compound. The carbonyl group (C=O) characteristic of carboxylic acids was observed within the range of 1725–1700 cm^{-1}. Due to conjugation with an aromatic ring, this absorption is shifted to a lower frequency range of 1700–1680 cm^{-1}, which is typical for such structures . The para-substituted aromatic ring is evidenced by bending vibrations at 842 cm^{-1}. The absorption band at 1599 cm^{-1} indicates the presence of a C=C stretching vibration. Additional bands at 1573 cm^{-1}, 1513 cm^{-1}, and 1424 cm^{-1} further support the presence of aromatic C=C bending vibrations. Moreover, the broad and intense O–H stretching absorption observed in the range of 3300–2536 cm^{-1} is attributed to strong hydrogen bonding typical of carboxylic acid groups

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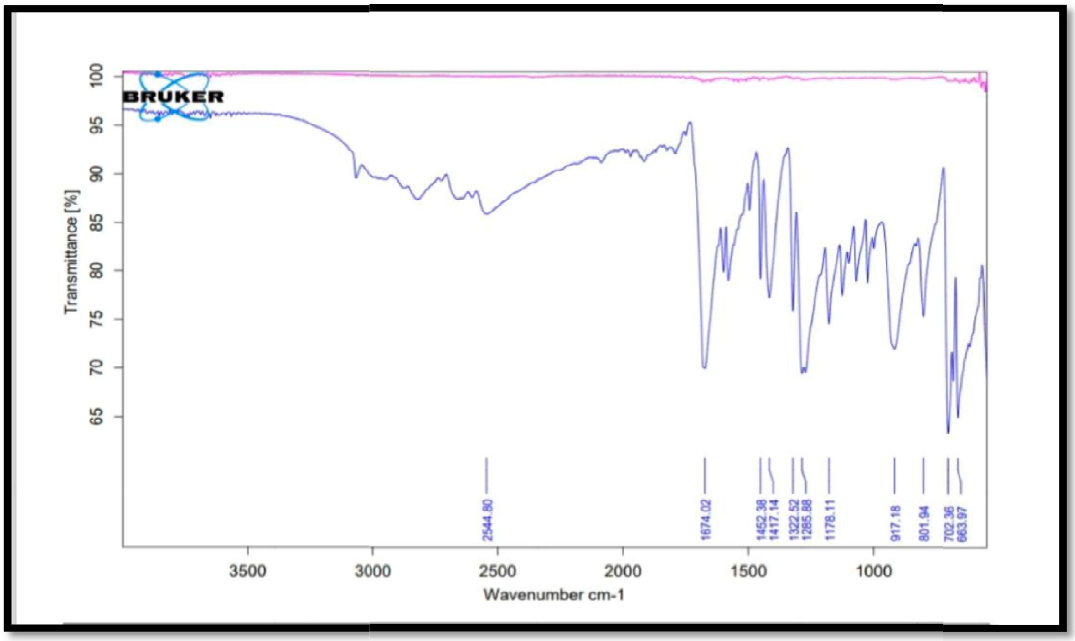
The resulting spectrum is a plot of the intensity of light absorbed by the sample as a function of wavelength, which is referred to as an infrared spectrum. FTIR can be used to analyze a wide range of samples, including gases, liquids Solids and powders. It is a powerful tool for identifying unknown compounds, monitoring chemical reactions, and characterizing materials. The technique is relatively fast, non- applications. overall, FTIR spectroscopy is a valuable analytical technique that provides important information about the chemical composition and structure of materials. applications makes it an essential tool in many fields, an essential tool in many fields, and its ease of use and reliability make it a popular choice among researchers and practitioners.

Fourier transform spectroscopy is a less intuitive way to obtain the same information. Rather than shining a monochromatic beam of light (a beam composed of only a single wavelength) at the sample, this technique shines a beam containing many frequencies of light at once and measures how much of that beam is absorbed by the sample. Next, the beam is modified to contain a different combination of frequencies, giving a second data point. This process is rapidly repeated many times over a short time span. Afterwards, a computer takes all this data and works backward to infer what the absorption is at each wavelength.

The beam described above is generated by starting with a broadband light source—one containing the full spectrum of wavelengths to be measured. The light shines into a Michelson interferometer—a certain configuration of mirrors, one of which is moved by a motor. As this mirror moves, each wavelength of light in the beam is periodically blocked, transmitted, blocked, transmitted, by the interferometer, due to wave interference. Different wavelengths are modulated at different rates, so that at each moment or mirror position the beam coming out of the interferometer has a different spectrum.

As mentioned, computer processing is required to turn the raw data (light absorption for each mirror position) into the desired result (light absorption for each wavelength).[2] The processing required turns out to be a common algorithm called the Fourier transform. The Fourier transform converts one domain (in this case displacement of the mirror in cm) into its inverse domain (wavenumbers in cm−1). The raw data is called an "interferogram"



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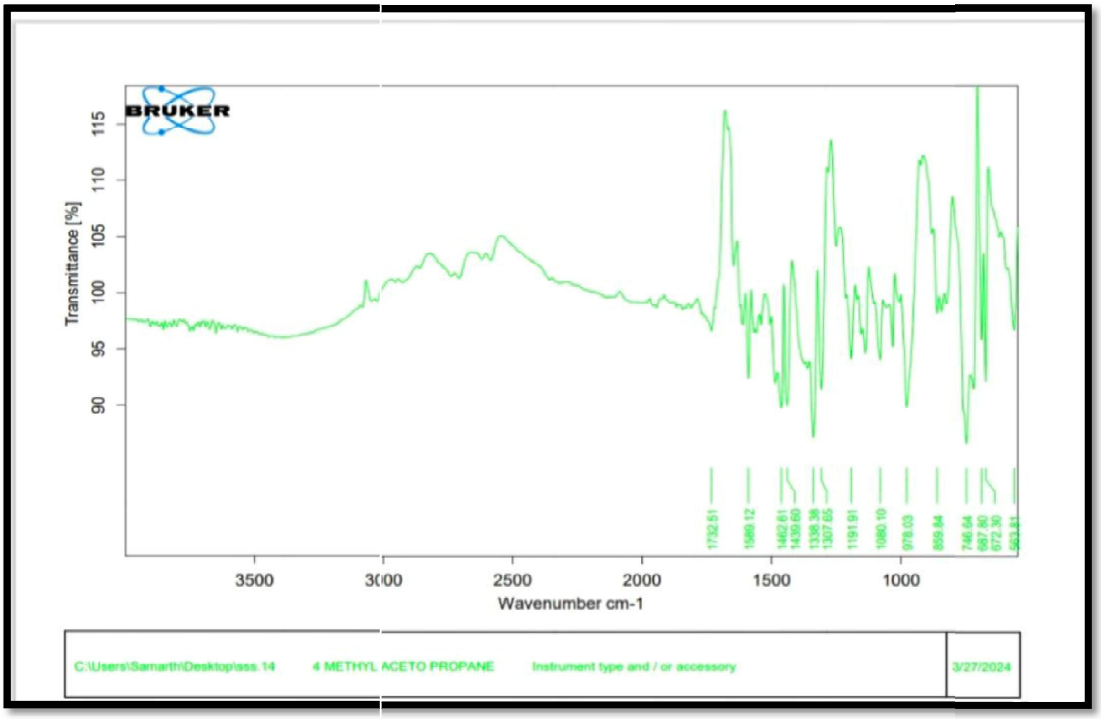

Fig.1.5 FTIR P methlycinnamic acid derivatives

NMR Spectroscopy:

Nuclear Magnetic Resonance (NMR) is a powerful analytical technique used to investigate the structure, composition, and dynamics of molecules. NMR is based on the interaction between atomic nuclei and a strong magnetic field and radiofrequency radiation. It is widely used in various fields, including chemistry, biochemistry, materials science, and medicine. In NMR, a sample is placed in a strong magnetic field and then subjected to radiofrequency radiation. The nuclei in the sample absorb and re-emit the radiation at a characteristic frequency, which depends on the local environment of the nucleus. By detecting the frequency of the emitted radiation, NMR can provide detailed information about the molecular structure and composition of the sample.

NMR can provide a wealth of information about a sample, including the number and types of atoms, the chemical environment of each atom, and the connectivity of atoms within a molecule. This information can be used to determine the identities' trans-configuration is confirmed by a large coupling constant (~15 -16 Hz)for the Olefinic protons in 'H NMR.The presence of a singlet at ~2.35 ppm confirms the para-methyl Group on the phenyl ring.Downfield shift of carboxylic carbon(~170 ppm) is characteristic Of cinnamic acid derivatives in I9C NMR.

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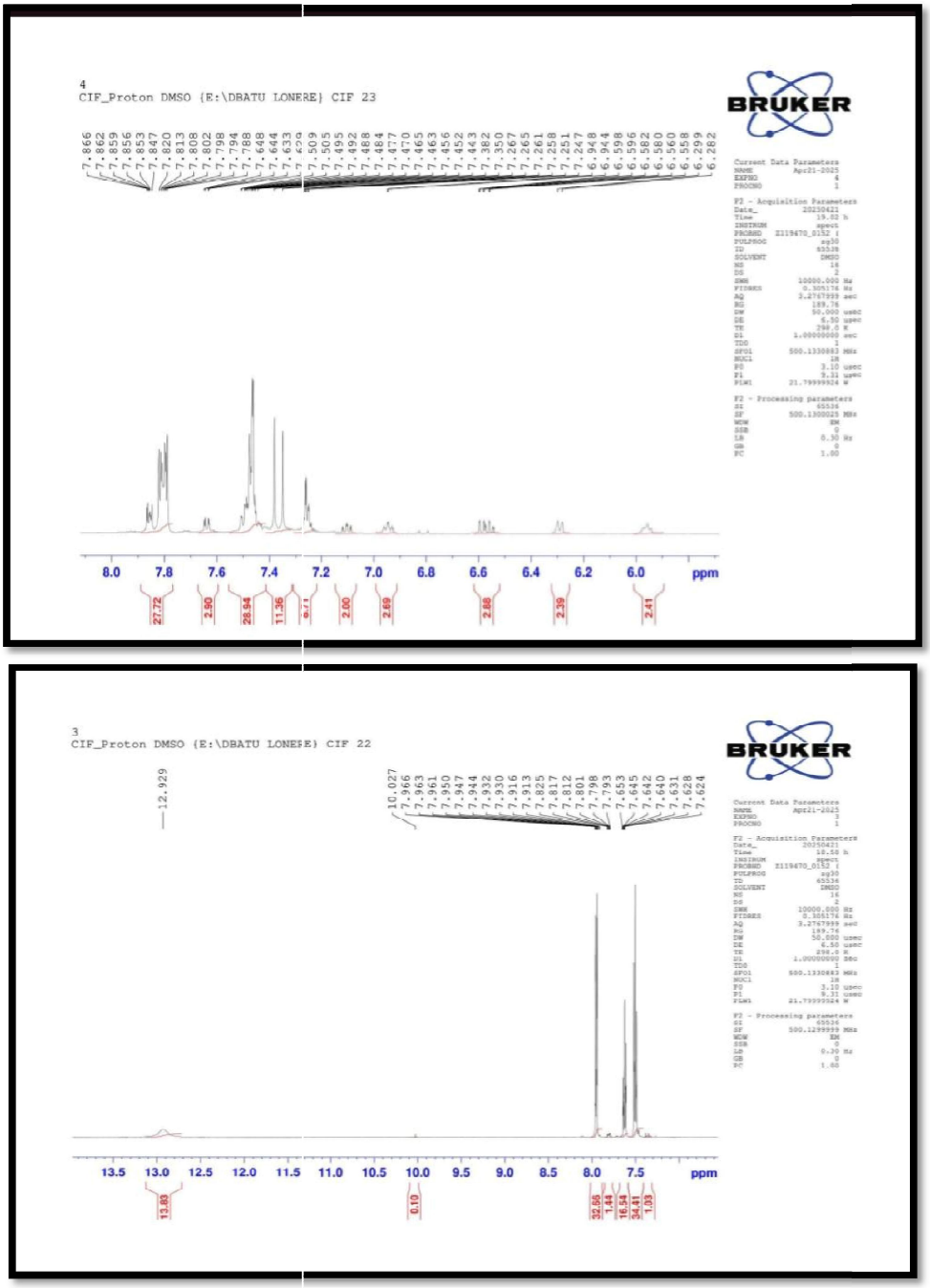

Fig: 1.6 NMR Spectroscopy of P methlycinnamic acid

Proton Type	δ Range (ppm)	Multiplicity	Coupling Constant (J)	Integration	Notes
Ar-H (aromatic protons)	7.20 – 7.40	Multiplet	–	4H	Para-substituted pattern

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Proton Type	δ Range (ppm)	Multiplicity	Coupling Constant (J)	Integration	Notes
CH=CH (olefinic protons)	6.30 – 7.00	Doublet	~15–16 Hz	2H	Trans coupling
CH_3 (para to phenyl ring)	2.30 – 2.45	Singlet	–	3H	p-Methyl group

Fig: 1.7 ^{13}C NMR (in $CDCl_3$, δ ppm)

Carbon Type	δ Range (ppm)	Notes
COOH (carboxylic acid)	~167 – 173	Highly deshielded
Olefinic C=C (β-carbon)	~118 – 145	Two distinct peaks
Aromatic Carbons	~125 – 140	Typical aryl shifts
CH_3 (aromatic methyl group)	~20 – 25	Shielded region

Fig: 1.7 ^{1}H NMR (in $CDCl_3$, δ ppm)

CHROMATOGRAPHIC ANALYSIS:

Thin layer chromatography (TLC) is a type of chromatography used to separate and identify different components of a mixture. It involves placing a small spot of the sample on a thin layer of adsorbent material, such as silica gel or alumina, which is coated on a glass or plastic plate. The plate is then placed in a solvent, which travels up the plate by capillary action, carrying the components of the sample along with it. As the solvent moves up the plate, the different components of the sample are separated based on their affinity for the adsorbent material and the solvent. Components that have a strong affinity for the adsorbent material will move more slowly up the plate, while those with a weaker affinity will move faster. This results in the compo of the sample separating out into distinct bands or spots on the plate. Once the separate is complete, the plate is removed from the solvent and dried. The spots or bands can then be visualized using various techniques, such as staining with a chemical reagent or exposing the plate to UV light. The Rf value, or retardation factor, can be calculated for each component, which is the ratio of the distance traveled by the component to the distance traveled by the solvent. The Rf value is a characteristic property of a compound and can be used to identify unknown compounds by comparing their Rf values to those of known compounds. Silica gel G acted as stationary phase whereas the following solvent systems were used as mobile phase.

Chloroform: Methanol (9:1)

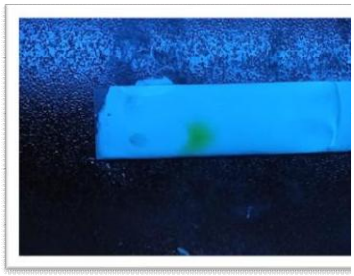

Fig. A :- TLC Plate Of P methlycinnamic acid

Fig: B TLC Plate Of benzaldehyde and p methlycinnamic acid And P Tolualdehyde

The Rf value is the ratio of the distance traveled by the component to the distance traveled by the solvent front. This value is characteristic of each component and can be used to identify the components of the mixture.

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Retention Factor (RF) = Distance travelled by the component
Distance travelled by the solvent.

The retention factors are as follows:

Sr.	Figure	Rf Value
1	Fig A	0.25-0.35
2	Fig B	0.30-0.45

BIOLOGICAL ACTIVITY:

Liquid dilution method or Test tube method

Use a series of test tubes which contain a double-strength medium and are labelled as Test tube numbers. In the first tube (un-inoculated), inoculum is not added which is used for checking the sterility of the medium. All other in a eleven test tubes, inoculum (3 to 4 drops) is added to reach the final concentration of microorganisms is 10 to 10 cells/ml. In all test tubes, test chemical is added ranging from 0.5 to 5 ml except in the control tube. The second tube (control) is used to check the suitability of the medium for growth of the test microorganism and the viability of the inoculum. The final volume min all test tubes iadjusted by using sterile water. The contents of all test tubes a properly mixed and incubated at 37°C for 2 to 3 days. After incubation, all test tubes an examined for the growth in the form of turbidity and the results are recorded and minim inhibitory concentration is calculated. It is also necessary to conduct a preliminary experiment to determine the approximate range (test solution) which would be suitable the test.

Antimicrobial Activity:

The inhibition zone diameter of all synthesized compounds was determined through the cup- plate agar diffusion method to evaluate their in vitro antimicrobial activity against Staphy/ococcus aureus using p methlycinnamic acid derivative product dissolved in methanol and placed in right side cup as a test and methanol as control in left side cup. A clear ring surrounding the antimicrobial source where bacterial growth is prevented.

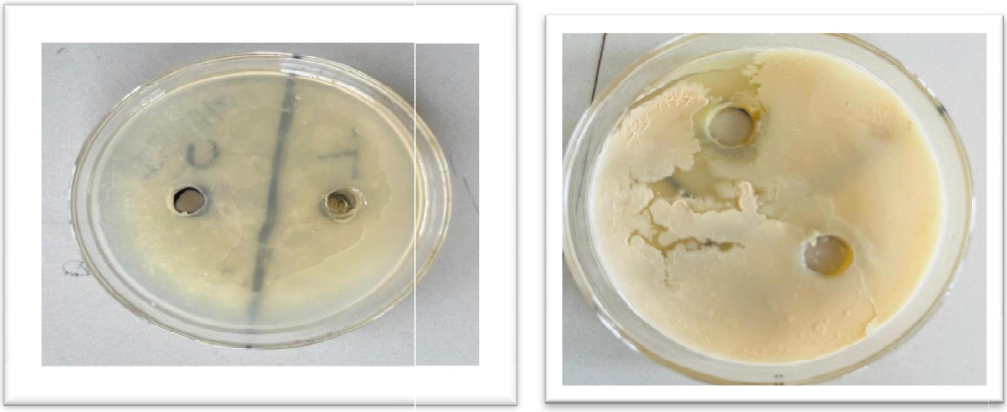

Fig: 1.6 Antimicrobial Screening

Result:

To synthesize p-methlycinnamic via an aldol-type Condensation, p-tolualdehyde(4-methylbenzaldehyde) can be used as the starting aromatic aldehyde.Thermal decarboxylation of The intermediate-carboxylic acid yields-methylcinnamic acid's-Tolualdehyde (1equip) and malonic acid(1.2 -1.5 equip) were dissolved In pyridine (or ethanol)Yield Typically 60 -80%.depending on purification efficiency and reaction conditions The reaction afforded p-methylcinnamic acid in yields typically ranging from 70-85%.The product was characterized by melting point determination, IR, and NMR spectroscopy, confirming the E-configuration of the double bond.

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II. CONCLUSION

The fundamental goal of medicinal chemistry is the development of new anti microbial therapeutic agents. In conclusion, synthesis, characterization, and pharmacological activity of newly synthesized derivatives of p methlycinnamic acid are promising areas of research with a wide range of potential applications in the fields of organic chemistry and drug discovery. synthesis has been shown to be a rapid and efficient method for synthesizing new compounds, while characterization techniques such as FT-IR, 1H-NMR, and mass spectroscopy can provide valuable information on the molecular structure and purity of the

The compound p-methoxy cinnamic acid (PMCA) can be synthesized by The Perkin reaction with assisted ultrasonic waves. This method requires a longer time for PMCA synthesis. Simple GIAO calculations allow to rationalize the Splatting's observed for ortho and meta carbons of cinnamic acids in the solid state. In solution, the free rotation about the single bonds linking the phenyl ring to CH-CH-CO, R and X substituents suppresses the splitting averaging the signals. Moreover, the calculations allow an assignment of the spitted signals, thus offering an alternative to a problem of great experimental difficulty since it requires very Given its dual antibacterial and anti-inflammatory properties, p-methylcinnamic acid holds potential as a lead compound in the development of multifunctional therapeutic agents. Further in-depth pharmacokinetic studies and clinical evaluations are warranted to validate its efficacy and safety profiles.

ACKNOWLEDGEMENT

I sincerely express my gratitude to Dr Gaikwad Shital D., Department of Pharmaceutical Chemistry Samarth Institute of Pharmacy, Belhe. for their valuable guidance, constant encouragement, and support throughout the course of this Project.I would also like to thank the Principal and Staff Members of the Department of Pharmacy for providing the necessary facilities and resources to carry out this research work. Special thanks to my family and friends for their moral support, and to all those who helped directly or indirectly in the completion of this project.

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