Synthesis, Eugenoxyacetic Acid Structure and Some Exports

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Abstract
Successfully synthesized acid structure from *Ocimum tenuiflorum* essential oil and monochloroacetic acid. Their structures were elucidated by 1H – NMR and 13C–NMR nuclear magnetic resonance spectroscopy. Through investigation, the reaction achieved the highest efficiency with the molar ratio between eugenol and monocloacetic acid 1:1,2 and the reaction temperature from 100 - 1050C.

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1. Introduction

The Vienammese is located in a tropical climate zone, which is blessed with an incredibly rich natural environment, home to many valuable medicinal plants. Eugenoxyacetic acid is a growth stimulant that has gained particular attention due to its advantages in both usage and synthesis [1-5].

Recent studies have reported that eugenocyacetic acid stimulates seed germination in rice, corn, green beans, and potatoes, as well as root and shoot growth in crops like tomatoes, cucumbers, watermelons, rice, corn, and green beans [6, 9]. Furthermore, synthetic eugenoxyacetic acid, derived from eugenol extracted from basil essential oil, is non-toxic to the environment and human health. This is in line with the global trend of research focusing on human health. Therefore, this study was conducted to synthesize eugenoxyacetic acid and some derivatives, while determining the structure of eugenoxyacetic acid and investigating factors influencing the reaction efficiency (molar ratio, reaction temperature) [10-14].

Experimental

Equipment

^{1}H-NMR spectra were measured on a Bruker Avance 500 MHz spectrometer, using methanol as the solvent. 13C-NMR spectra combined with DEPT-NMR were measured on a Bruker Avance 500 MHz spectrometer, using methanol as the solvent.

Chemicals

Basil essential oil (eugenol ~70%), monochloroacetic acid, toluene, NaOH, ethyl acetate, acetic acid (CH_3COOH), ethanol (C_2H_5OH), HCl, and methanol (CH_3OH).

Method

Synthesis of Eugenoxyacetic acid

Dissolve 94.5 grams (1 mol) of monochloroacetic acid in 150 ml of water, then slowly add Na2CO3 until gas bubbles cease. This will produce solution A. Dissolve 75 grams of NaOH in 200 ml of water, then add 200 ml of basil essential oil (approximately 70% eugenol) to produce solution B. Mix solutions A and B, then heat the mixture under reflux while stirring for 2.5-3 hours. After the reaction is complete, acidify the mixture with HCl to obtain eugenoxyacetic acid in the form of a yellow solid. Recrystallize in water to obtain fine white needle-shaped crystals. The crystals are then recrystallized in toluene.

Results and Discussion

Synthesis of eugenoxyacetic acid

The synthesis of eugenoxyacetic acid was carried out according to the Williamson reaction. In this reaction, eugenol reacts with monochloroacetic acid in a basic medium.

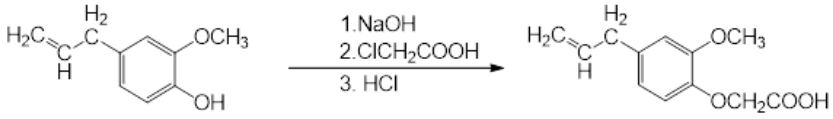


Fig 3.1: Eugenoxyacetic acid obtained in the laboratory of the Department of Chemistry, Tay Nguyen University

Structural identification results

The ^{1}H-NMR and 13C-NMR spectra of eugenoxyacetic acid are shown in Figures 3.2 and 3.3. The structural elucidation of the compound is presented in Table 3.1.

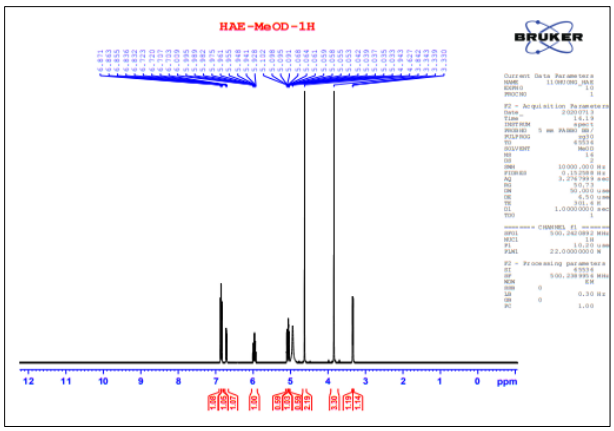


Fig 3.2: 1H NMR spectrum of eugenoxyacetic acid

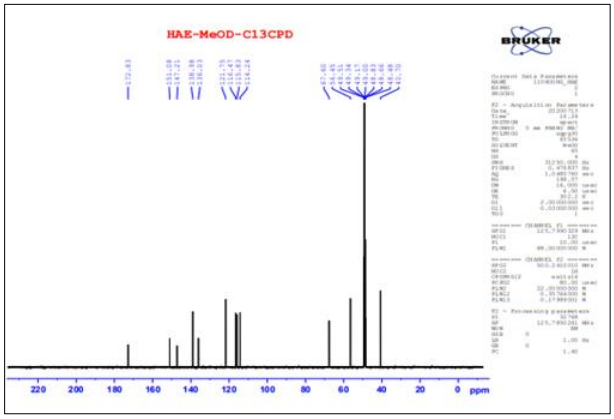


Fig 3.3: 13C NMR Spectrum of Eugenoxyacetic Acid

Table 3.1: Structural Elucidation of Eugenoxyacetic Acid

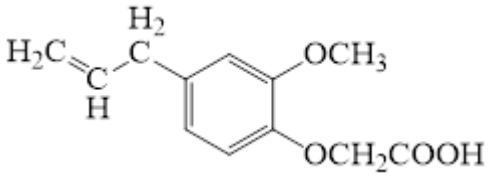
CH_n	δ_C (ppm)	δ_H m, (J,Hz), nH	Note
COOH	161.2	-	
C	158.4	-	
C	152.7	-	
C	149.6	-	
CH	144.8	7,59d (2,5Hz), 1H	
CH	139.3	8,15d (9,5Hz), 1H	
C	112.7	-	
CH	112.6	6,27d (9,5Hz), 1H	
C	106.5	-	
CH	105.0	7,02s, 1H	
CH	93.9	7,14s, 1H	
O-CH_3	60.1	4,27 s, 3H	

The compound isolated shows the following spectral data:

a) **^{13}C-CPD Spectrum:** Displays signals for 12 carbon atoms, including a signal at δ_C(max) = 161.2 ppm for the -COOH group.
- A signal for -OCH3 group.
- A CH2 signal is also present.

b) **1H-NMR Spectrum:** Displays signals for 14 protons.
- No signal for the –OH group of the aromatic ring.
- The signal at δ_H = 4.27 ppm corresponds to a 3H signal, which is attributed to the -O-CH_3 group.

By comparing the 1H NMR and 13C NMR spectra of eugenoxyacetic acid, it can be concluded that the isolated compound has the following structure [15]:
This conclusion is based on the spectral data analysis which matches with the known structure of eugenoxyacetic acid.



Investigation of factors affecting yield

Investigation of the molar ratio between eugenol and monochloroacetic acid.

One of the factors affecting the reaction yield is the molar ratio between eugenol and monochloroacetic acid. Therefore, we conducted an investigation on this factor to determine the optimal molar ratio for the highest yield. The results are presented in Table 3.2.

Table 3.2: Investigation of the molar ratio between eugenol and monochloroacetic acid.

STT	Eugenol (mol)	Acid monocloacetic	Efficiency (%)
1	0,1	0,09	78,0
2	0,1	0,10	86,0
3	0,1	0,11	89,0
4	0,1	0,12	90,0
5	0,1	0,13	89,0
6	0,1	0,14	90,0
7	0,1	0,15	90,0

Investigation of the reactant ratio:

The investigation of the reactant ratio showed that the molar ratio between eugenol and monochloroacetic acid of 1:1.2 resulted in the highest yield of eugenoxyacetic acid, reaching 90%. In contrast, when the molar ratio was 1:1, the yield of the synthesized product was only 86%. Therefore, for the subsequent stage, we selected the 1:1.2 ratio.

Investigation of the effect of temperature:

We also investigated the effect of temperature on the reaction, using a molar ratio of eugenol to monochloroacetic acid of 1:1.2, with a reaction time of 180 minutes. The reaction temperatures ranged from 80°C to 105°C.

Table 3.3: The results are presented in

No	(^{0}C)	H (%)
1	80	70,0
2	85	81,0
3	90	85,5
4	95	89,0
5	100	91,0
6	105	90,8

Conclusion

The synthesis and structural determination of eugenoxyacetic acid from eugenol (derived from essential oil of *Ocimum basilicum*) and monochloroacetic acid were successfully carried out using nuclear magnetic resonance (NMR) techniques.

We also investigated the molar ratio and the impact of temperature, showing that the optimal reaction conditions were achieved with a molar ratio of eugenol to monochloroacetic acid of 1:1.2, and a reaction temperature between 100°C and 105°C, which provided the highest yield. We will continue to explore the biological activities of the synthesized compound and aim to synthesize further derivatives of eugenoxyacetic acid from eugenol.

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References

1. Arya SP, Bhatia SC, Bansal A. Extractive spectrophotometric determination of tin as Sn(II)-ferron complex. Fresenius J Anal Chem. 1993;345(11):679-82.
2. Awasthi P, et al. Eugenol derivatives as future potential drugs. J Pharm Res. 2008;1:215–20.
3. Berg OV, Jager WF, et al. A wavelength shifting fluorescent probe for investigating physical aging. Macromolecules. 2006;39(1):224-31.
4. Berg OV, Jager WF, et al. 7-Dialkylamino-1-alkylquinolinium salts: highly versatile and stable fluorescent probes. J Org Chem. 2006;71:2666-76.
5. Ministry of Health. Vietnamese Pharmacopoeia I. Vol. 2. Hanoi: Medical Publishing House; 2002. p. 383–4.
6. Carrasco HA, et al. Eugenol and its synthetic analogues inhibit cell growth of human cancer cells (part I). J Braz Chem Soc. 2008;19:543–8.
7. Deraeve C, Boldron C, et al. Preparation and study of new poly-8-hydroxyquinoline chelators for an anti-Alzheimer strategy. Chem Eur J. 2008;14:682–96.
8. Crousse B, Bégué JP, et al. Synthesis of tetrahydroquinoline derivatives from α-CF3-N-arylaldimine and vinyl ethers. Tetrahedron Lett. 1998;39:5765–8.
9. Daniel KG, Gupta P, et al. Organic copper complexes as a new class of proteasome inhibitors and apoptosis inducers in human cancer cells. Biochem Pharmacol. 2004;67:1139–51.
10. Fortuna CG, Barresi V, et al. Synthesis and NLO properties of new trans-2-(thiophen-2-yl)vinylheteroaromatic iodides. Arkivoc. 2009;8:122–9.
11. Fournet A, Barrios AA, et al. Antiprotozoal activity of quinoline alkaloids isolated from Galìpea longiflora, a Bolivian plant used as a treatment for cutaneous leishmaniasis. Phytother Res. 1994;8:174–8.
12. Do HI, Dang QC, Bui XC, Nguyen TD, Do TD, Pham VH, Vu NL, Pham DM, Pham KM, Doan TT, Nguyen T. Medicinal Plants and Animals Used in Vietnam. Vol. 1. Hanoi: Institute of Medicinal Materials; 2003. p. 1027–9.
13. Rojo L, et al. Eugenol functionalized poly(acrylic acid) derivatives in the formation of glass-ionomer cements. Dent Mater. 2008;24:1709–16.
14. Sadeghian H, et al. Design and synthesis of eugenol derivatives, as potent 15-lipoxygenase inhibitors. Bioorg Med Chem. 2008;16:890–901.
15. Hoang DX. A study on the synthesis, structure, and properties of several derivatives of eugenoxyacetic acid and isoeugenoxyacetic acid [master's thesis]. Hanoi: Hanoi National University of Education; 2006.