

14TH ASIAN CHEMICAL CONGRESS 2011

CONTEMPORARY CHEMISTRY FOR SUSTAINABILITY AND ECONOMIC SUFFICIENCY

5-8 SEPTEMBER 2011
BANGKOK, THAILAND

[WWW.14ACC.ORG](http://www.14acc.org)



HOSTED BY CO-HOSTED BY



THE AUSPICES OF



SUPPORTED BY



14ACC International Advisory Committee

Prof. Jung-Il Jin

Past-President, IUPAC, Korea University, Korea

Prof. David StC. Black

Secretary General, IUPAC, University of New South Wales, Australia

Prof. Chunli Bai

President, FACS, Key Laboratory of Molecular Nanostructure and Nanotechnology, Institute of Chemistry, the Chinese Academy of Sciences, China

Prof. Datuk Ting-Kueh Soon

Immediate Past-President, FACS, Vice President, Institute Kimia Malaysia, Malaysia

Prof. Long Lu

Secretary General, FACS, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, China

Prof. San H. Thang

Treasurer, FACS; Senior Principal Research Scientist, CSIRO Molecular and Health Technologies, Australia

Prof. Tahsin J. Chow

Coordinator of Projects, FACS, Institute of Chemistry, Academia Sinica, Chemical Society located in Taipei

Prof. Kyung Byung Yoon

Director of Scientific Affairs, FACS, Sogang University, Korea

Prof. Noriyuki Suzuki

Editor of Publication, FACS, Japan

Prof. Guoqiang Lin

Shanghai Institute of Organic Chemistry, CAS, China

Prof. Minoru Isebe

National Tsing Hua University, Taiwan

Prof. Thomas H. Lane

Past-President, ACS, USA

Prof. Nancy B. Jackson

President, ACS, USA

Prof. Timothy Deming

Professor, Department of Bioengineering University of California, Los Angeles, USA

Prof. Yongyuth Yuthavong

NSTDA, Thailand

Prof. M.R. Jisnuson Svasti

President, the Science Society of Thailand

Prof. Somsak Rujirawatana

Chulabhorn Research Institute, CRI, Thailand

Prof. Apichart Suksamran

Ramkhamhang University, Thailand

Content

Code	Title	Page
OR-G1-11	The Biodegradable Polymer Electrolyte from Cellulose Acetate for Lithium Ion Battery Application	1
OR-G1-18	A Reinforcement for beta-Chitin Films from Banana Peels Cellulose Microfibril	8
OR-G1-21	Sol-Gel Preparation of ZnO/HDTMA/Hectorite and Its Adsorptive Photocatalytic Study Towards Alizarin Red S Degradation	14
PO-G1-21	Antibacterial Activity of Encapsulated Thymol	21
PO-G1-22	The Effect of Coffee Residue on Cure Characteristics and Physical Properties of Natural Rubber	26
PO-G1-23	Effect of Molecular Weight of Dispersing Agent on Mechanical and Thermal Properties of Natural Rubber/Silica Nanocomposites	33
PO-G1-24	Chemical and Physical Characteristics of Heat-Treated Leonardite	40
PO-G1-25	Encapsulated N-Eicosane as Phase Change Material	46
PO-G1-38	Electrochemical Behaviors of PEO Based Electrolytes Containing Imidazolium Salt Modified Inorganic Fillers	52
PO-G1-41	Synthesis and Analysis of Polyaniline/TiO ₂ Composites Prepared with Various Concentrations of p-TSA	58
PO-G1-42	Effect of Poly(Styrene Sulfonic Acid) Concentration on Electroactivity of Polyaniline/Platinum Composites	64
PO-G1-43	Anionic Surfactant Sensor Based on Polydiacetylenes Containing Amino Group	70
PO-G1-54	Synthesis and Characterization of ZnO Thin Films Prepared by Sol-Gel Method	77
OR-G2-12	Solid Phase Extraction of Gold(I) Thiosulfate by Cetyl Trimethyl Ammonium Bromide (CTAB) on Nata de Coco Membrane	82
OR-G2-18a	Quantitative Analysis of 16 Priority PAHs in the Major Rivers of Southern Border Provinces of Thailand	88
PO-G2-03	Rapid Determination of Ferulic Acid and Total Antioxidant Using Lab-On-Paper with Dual Electrochemical and Colorimetric Detection	93
PO-G2-09	Method Development for Preconcentration and Determination of Lead and Cadmium by Sequential Injection-Anodic Stripping Voltammetry Using Bismuth Film Screen-Printed Carbon Nanotube Electrode	100

Code	Title	Page
PO-G2-12	Parabens Determination by Ultra-Performance Liquid Chromatography Coupled with Electrochemical Detection	106
PO-G2-23	Preparation of Sewage Sludge-Based Adsorbent for Ammonia Gas Removal	112
PO-G2-39	Liquid Chromatographic Determination of Scopolamine in Hair with Free-Swimming Single Drop Liquid Phase Microextraction Technique	119
PO-G2-54	Two New Fluorescent Macrocycles Derived from a Novel Dialdehyde: A Novel Fluorescent Chemosensor for Zinc Ion	129
PO-G2-55	Greener Anodic Stripping Voltammetric Method Employing In Situ Plated Bismuth Film Electrode for Determination of Cadmium and Lead	135
PO-G2-75	Liquid Chromatography with C8-Silica Monolith in a Microchip	141
OR-G3-04	A Laboratory on Kinetics of Reaction between Iodate and Bisulfite for Secondary Students	146
OR-G4-06	Synthesis, Characterization and Emission Properties of Yttrium(III) and Lanthanide(III) Complexes of Tripodal Heptadentate Schiff-Base Ligands	152
OR-G4-19	Antimicrobial and Photocatalytic Activity of ZnO Nanostructures	158
OR-G4-20	NiFe ₂ O ₄ Catalyst: The Influence of Gas Feed Composition on CO ₂ /H ₂ Conversion into Alcohol Compounds	167
PO-G4-05	Effect of Calcination Temperature on Crystal Structure and Morphology of LaCoO ₃ Powder Prepared by Mechanochemical Method	173
PO-G4-07	Study on the Interaction between Curcumin with d ¹⁰ Metal Ion	179
PO-G4-15	RuCl ₃ .nH ₂ O: A Valuable Catalyst for One-Pot Synthesis of Hormone Steroid Derivatives	185
PO-G4-31	Synthesis and Characterization of Mesoporous CoFe ₂ O ₄	190
PO-G4-33	Hydrothermal Synthesis of Calcium Titanate Nanostructures	197
PO-G4-53	Hg(II)-Selective Fluoroionophoric Behaviors of a 2-(3-(2-Aminoethylsulfanyl) Propylsulfanyl)Ethanamine Bearing a Naphthalimide Fluorophore	203
PO-G4-84	Structural Property of Copper Phosphates Studied by X-ray Absorption Spectroscopy and Their Catalytic Property	209
PO-G4-88	Microwave Synthesis of Nano-Zeolite a From Rice Husk Ash	215

Code	Title	Page
OR-G5-06	Transformation of 4-Allylpyrocatechol Diacetate into Chavibetol in Vietnam Betel Leaves (<i>Piper betle</i> L.)	220
OR-G5-12	Toxicity of Stemonon Extract and Detoxification Enzymes Activity in <i>Spodoptera litura</i> (F.)	228
OR-G5-14	Evaluation of Antihepatotoxic Activity of <i>Berberis pachycantha</i> Koehne. Rhizome in Carbon Tetrachloride Induced Toxicity	234
OR-G5-15b	Solid Phase Peptide Synthesis of Some Analogs of Bradykinin Hormone Using Microwave Energy Application (Part II)	240
PO-G5-08	Chemical Constituents and Antimicrobial of Crude Extracts from <i>Xylaria sp. Fungi</i>	245
PO-G5-25	Chemical Constituents of the Essential Oils of White Tumeric (<i>Curcuma zedoria</i> (Berg.) Roscoe) from Indonesia and Its Toxicity Toward <i>Artemia Salina</i> Leach	248
PO-G5-37	Vanillin Structure Modification of Isolated Vanilla Fruit (<i>Vanilla Planifolia</i> Andrews) to form Vanillinacetone	252
PO-G5-38	Application of Statistical Experiment Design for Optimization of Protease Extraction from Horse Mango (<i>Mangifera foetida</i> Lour) Kernel	259
PO-G5-39	Isolation and Identification of p-Hydroxybenzaldehyde from <i>Bambusa vulgaris</i> Schrad. Shoots as a Marker Compound for Standardization of Traditional Medicine	265
PO-G5-41	Determination of Prednisolone in Thai Traditional Medicine Types: Bolus and Drug Powder Selling in Nakhon Pathom, Thailand	271
OR-G6-01a	Production of Ammonia by <i>In-Situ</i> Hydrolysis of Urea for Safe Feedstock in Power Plants	276
PO-G6-24	Synthesis of Phthalimides, Phthlamidic Acids and Related Compounds under Green & Eco-Friendly Conditions	283
OR-G7-15	Theoretical Studies on the Effect of Anchoring Group in D-D- π -A Organic Dyes for Dye-Sensitized Solar Cells (DSCs)	288
PO-G7-04	An Antitubercular Agents Based on Quantum Calculations	296
PO-G7-05	Elucidating the Potential Binding Mode of Calanolide a Derivatives in WT and Y181C HIV-1 RTs Based on Molecular Modelling	301
PO-G7-12	A Computational Raid to Structure and Bonding of $Cp_2Ti(C_6H_4-nF_n)$ ($C_6H_4=$ Benzyne, $n=1-4$) Complexes	306
PO-G7-18	Study of Eg5 Allosteric Protein Slowed Down by STLC Inhibitor Using Computational Approach	314

Code	Title	Page
PO-G7-19	Theoretical Investigation of Organic Dyes Containing Carbazole-Diphenylamine-Benzene-Cyanoacrylic Acid for Dye-Sensitized Solar Cells	319
PO-G7-27	Molecular Modeling and Quantum Chemical Calculations of High Potency Anti-Tuberculosis Agents in Class of Triclosan and Diphenyl Ether Derivatives as InhA Enzyme of Mycobacterium tuberculosis	326
OR-G8-02	Performance of Black Powder Using Different Charcoal Types as Propellant for Fireworks Aerial Shells: an Assessment	333
OR-G8-06	Analysis of Bed Voidage Characteristic of a Gas-liquid-solid Fluidized Bed by CFD Simulation and Experiment	339
PO-G8-05	Design of a Vibration Type Coiled Tube Heat Exchanger for Hard Chrome Plating Process	347
PO-G8-06	Effects of Conducting Polymer in GEL-AGM-VRLA Battery	352
PO-G8-07	Corrosion Condition Study of Steel and Stainless Steel Pipes by Ethanol in Ethanol Production Industry	358
PO-G8-10	Optimization of Wax Esters Production from Palm Fatty Acid Distillate and Oleyl Alcohol Over Amberlyst 15 by Response Surface Methodology	366
PO-G8-11	Production of Free Fatty Acid from Hydrolysis of Waste Coconut Milk from Waste Water Pond Using Hydrochloric Acid	373
OR-S1-03	Carbon Dioxide Adsorption Capability of Steel-Making Slags	378
PO-S1-04	Utilization of Carbon Dioxide in Organic Synthesis Using Ni/CeO ₂ -ZrO ₂ Catalyst	384
OR-S3-02	Development and Validation of a HPLC Method for the Determination of Mitragynine in the Kratom Cocktail and the Extracted of Kratom Leaves (<i>Mitragyna speciosa</i> Korth.)	389
PO-S5-05	Development of Flow Injection Analysis with Modified Amperometric Biosensor for Determination of Glucose in Biological Fluids	399
OR-S11-01	Sensitization of N-Doped TiO ₂ Film by <i>In Situ</i> Adsorption Ruthenium Polipyridine Complexes for Dye-Sensitized Solar Cells	406
PO-S11-01	Synthesis and Characterization of the Ruthenium(II) Complex Containing 4,4'-Dicarboxy-2,2'-Bipyridine and 5-Methyl-2-(Phenylazo)Pyridine Ligands	412
PO-S11-02	Influence of Concentration and Type of Polyethylene Glycol on Structure of Porous TiO ₂ Photoelectrode for Dye-Sensitized Solar Cells	418

Code	Title	Page
PO-S11-03	Effect of Solvents Extraction of Achiote Seeds for Dye-Sensitized Solar Cells, Structural and Electronic Properties, Based on DFT Calculations	425
PO-S11-11	Catalytic Pyrolysis of Corncob Using Mesoporous Material	429
OR-S13-01	Chitosan Polyvinylchloride-Coated Wire Electrode for Lead Detection as an Environmental Sensor	436
OR-S13-04	Theoretical Study on Electronic Structure and Optical Properties of N-Coumarin Derivatives used as Oleds	443
PO-S20-01	Enhancing Photocatalytic Activity of Amorphous TiO ₂ by Doping with Anion	450
OR-G7-18	Comparative Molecular Field Analysis Study on Anti HIV-1 RT Diarylaniline Derivatives	460
OR-G7-19	3D-QSAR Study on Resveratrol Analogues as Aromatase Inhibitors	467
PO-S20-05	A Simple and Effective Method For Oxidation of Alcohols to Their Corresponding Carbonyl Compounds by The Use of Thallium (III) Nitrate Supported on Silica Gel	473
PO-G4-16	Degradation of Dye using Barium Titanium Oxalate Compound as Photocatalyst	478
PO-S19-03	The Synthesis of Aza-Anthraisoaxalone Derivatives as Cytostatic Compounds	484
INV-G7-16	Analytical Use of Ion Accelerators in Chemistry	490
INV0032	Nanoparticles as Carrier Systems for Topical Drug Delivery: Perspectives and Safety Aspects	498
OR-G6-04	Non-Catalytic Methyl Esterification of Variety Fatty Acids in a Bubble Column Reactor	504
PO-G3-04	Recent Developments of Catalysts for Green Friedel-Crafts Acylations	512
PO-S2-02	Co-Encapsulation of Curcumin and Ascorbyl Palmitate	518
OR-G4-06a	Photoelectrochemical Response of CuInS ₂ Films Electrode using RF Magnetron Sputtering	523
INV0105	Preparation of Ga-Doped AgIn ₅ S ₈ Electrodes using Chemical Bath Deposition for Photoelectrochemical Applications	531
PO-G1-58	Preparation of the Hybrid Material Ag/TiO ₂ /Bamboo Charcoal Composite by Sol-Gel Impregnation and Electrochemical Deposition Method	538

Vanillin Structure Modification of Isolated Vanilla Fruit (*Vanilla Planifolia* Andrews) to form Vanillinacetone

S. Handayani¹, R. Arianingrum¹ and W. Haryadi²

¹ Department of Chemical Education, Faculty of Mathematics and Natural Sciences, Yogyakarta State University, Karangmalang, Yogyakarta, Indonesia

² Department of Chemistry, Faculty of Mathematics and Natural Sciences, Gadjah Mada University, Bulaksumur, Yogyakarta, Indonesia
E-mail address: handayani137uny@yahoo.com

Abstract

The research has been conducted to isolate vanillin from Vanilla fruit (*Vanilla Planifolia* Andrews) and to modify its structure to form vanillinacetone. Generally, vanillin isolation was conducted by soxhlet extraction of vanilla fruit with ethylacetate as solvent, continued by hydrolysis with sodium hydroxide and acidic by hydrochloric acid. The resulting solution was then extracted with chloroform. The vanillin target has been separated by evaporation, and then purified by recrystallization from ethanol-water. Structure modification of vanillin was conducted through crossed aldol condensation between vanillin and excess acetone under basic condition to form vanillinacetone. The resulting material was characterized using FTIR spectrometer, ¹H and ¹³C NMR including 2D-NMR techniques (HMQC and HMBC).

Keywords: vanillin isolation, vanillinacetone, aldol condensation

1. Introduction

Vanilla (*Vanilla planifonia* Andrews) is one of natural source material availably overflowing in tropical area especially in Indonesia. In international market, Indonesian vanilla has known as *Java Vanila Beans*. The area of planting vanilla in Indonesia referred to the data of Directorate General of Plantation, Department of Agriculture, Republic Indonesia year 2008 is 25.429 ha [1]. Foreign exchange earnings through export of vanilla on year 2000 amounted to US \$31.4, overwhelmingly in the form of raw material. Therefore, the effort to increase the production of vanilla is always performed by practitioners of plantation researcher [2,3].

Vanilla fruit consists of two main parts, the peel of green fruit (60%) and white (20%), which plays an important role in the biosynthesis of vanillin. The vanillin content in the vanilla fruit is about 1.5 – 3 grams/100 of vanilla fruits. Minor compounds in the vanilla fruit are *p*-hydroxybenzaldehyde and *p*-methoxybenzyl methylether.

Vanillin could be obtained by synthesis, semi-synthesis and isolation from natural material. Vast biodiversity in Indonesia is more feasible and advantageous to obtain vanillin by isolation from natural materials. Vanillin could be isolated from vanilla fruit (*Vanilla planifolia* Andrews) with several methods such as enzymatic extraction [4], soxhlet extraction [5] and extraction using octylamine [6]. Therefore it still needs to develop the isolation of vanillin from vanilla fruit as basic material of the vanillin synthesis and structure modification to result more useful material.

Some benzalacetone derivatives [7], asymmetric dibenzalacetones [8] and hydroxydibenzalacetone [9] that were synthesized from benzaldehyde derivatives have been reported as antioxidant material. Vanillin as one of potential benzaldehyde derivative is expected to be modified to yield vanillinacetone.

2. Materials and Methods

2.1 Vanillin isolation

Procedure of soxhlet extraction was conducted as following: Fresh vanilla fruit that have been destructed were heated in the oven for 5 hours to reduce the water content before soxhlet extraction process. The extraction proceeds using ethylacetate and the circulation process was conducted for 25 times. Extract of ethylacetate was strained then evaporated with buchner subsequently and hydrolyzed with 1 N NaOH continued by adding HCl. The acidic solution was then extracted with chloroform to obtain vanillin which separated from the solvent by evaporation. The resulting powder was then purified by recrystallization. The physical properties such as colour, weight and melting point were determined.

2.2 Vanillin structure modification

0.02 mol NaOH was solved in 10 mL water, cooled with ice bath and 0.01 mol vanillin was slowly added under stirring until completely solved. 0.03 mol acetone was slowly dropped to the solution. The temperature was kept on room temperature during 3 hours stirring or until precipitant was produced. Afterward, 3 mL water was added before filtered using buchner and washed with water until pH below 7 was reached. The purified product was conducted using recrystallization method or gravitation column chromatography. The physical properties were determined based on its colour, weight and melting point. The purity test was conducted using TLC and TLC-scanner with some organic solvent. Structure elucidation was conducted using FTIR, ^1H and ^{13}C NMR, HMQC and HMBC. This procedure was repeated by substituted the solvent with ethanol-water and methanol and also replace the entering reagent method in to the reaction system to obtain the optimal result.

3. Results and Discussion

3.1 Vanillin isolation from *Vanilla planifolia* Andrews

Vanillin isolation from vanilla fruit was conducted using soxhlet extraction with ethylacetate. The result showed yield of 2.01 % with melting point of 81 °C and mild chocolate colour. The method provided good result since the vanillin content in vanilla fruit is around 1.5 – 3 % [4].

Generally, extraction and vanillin isolation from vanilla fruit was conducted through 3 steps : extraction using soxhlet extractor with ethylacetate continued by hydrolysis with strong acid and finally purified using recrystallization method. Hydrolysis process was conducted to break the glycoside binding between vanillin molecule and glucose as shown in Fig. 1. After hydrolysis process, acid was added to neutralize the remaining base. Hydrochloride acid was more efficient to neutralize the basic than sulfuric acid.

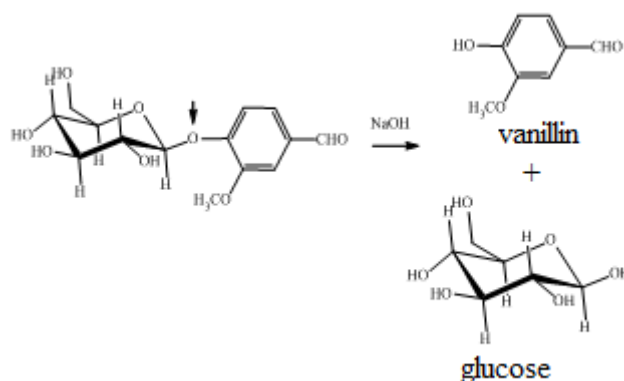


Figure 1. Hydrolysis reaction of vanillin glycosides

Based on standard vanillin melting point, it is known that vanillin melting point is 83°C, so it could be predicted that the vanillin isolation using ethyl acetate solvent resulting high purity of vanillin. The structure of isolated material was determined with spectroscopy, FTIR and NMR.

FTIR spectral data of isolated vanillin showed the existence of characteristic peak from aldehyde C–H stretching vibration at 2924 and 2854 cm^{-1} , whereas absorption at 1666 cm^{-1} showed stretching vibration of C carbonyl group. Strong absorption at 3186 cm^{-1} is characteristic of stretching vibration of O–H and at 1597 cm^{-1} and 1157 cm^{-1} refer to vibration of aromatic group and ether. It could be concluded from spectrum that resulting isolated material have functional group of aldehyde, aromatic ring, hydroxyl and ether. To elucidate the structure more detailed, then it was analyzed by two dimensional NMR and the data was shown at Table 1.

Table 1. NMR spectral data of isolated vanillin including HMBC and HMQC spectral data

No. C	$\delta\text{H}(\Sigma, m, J)$ (ppm)	δC (ppm)	HMBC (ppm)			
1'	-	130	-			
2'	7.417 (1H, s)	108.8	127.8	147	151	
3'	-	147	-			
3'-OMe	3.9 (3H, s)	56.3	151	147		
4'	-	151	-			
4'-OH	6.25 (1H, s)	-	114.5	147	151	
5'	7.04 (1H, d, 8.5)	114.5	130	127	147	
6'	7.43 (1H, d, 8.5)	127.8	108.8	151	191	
1	9.83 (1H, s)	191.15	108.8	130	147	151

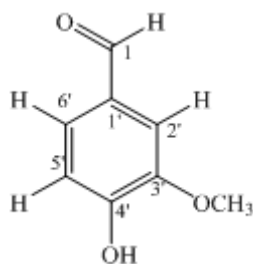


Figure 2. 4-hydroxy-2-methoxybenzaldehyde (Vanillin)

Based on the Table 1, it could be concluded that resulting isolated material from vanilla fruit is 4-hydroxy-2-methoxybenzaldehyde (vanillin) which showed in Fig. 2. This vanillin was then used as basic material of vanillin acetone synthesis.

3.2 Vanillin structure modification

The structure modification of vanillin was conducted using crossed aldol condensation reaction between vanillin with acetone and acetophenone (molar ratio 1:3). In this reaction, excess acetone was used to prevent the formation of dibenzalacetone as side product. The synthesis was highly solvent dependent so it used aquadest, ethanol-aquadest and methanol as solvent variation.

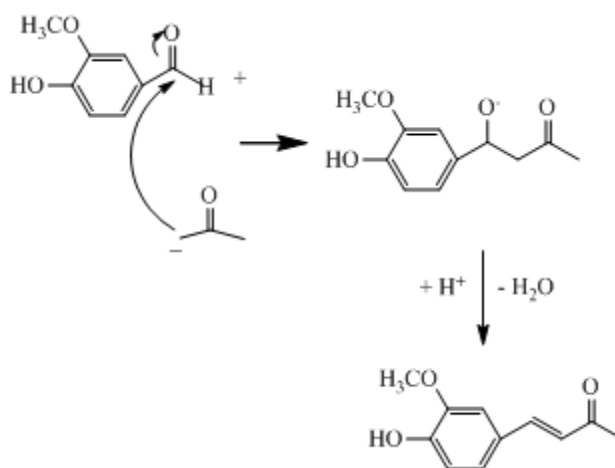


Figure 3. Scheme of vanillin structure modification

The reaction mechanism of the synthesis i.e. crossed aldol condensation that started by nucleophilic attack to C carbonyl as shown on Fig. 3. Nucleophile is compound that having $H\alpha$ in case of this reaction is acetone. Therefore, researcher started the aldol condensation by first mixing compound that have $H\alpha$ to form nucleophile [7,10]. Unfortunately, there is a disadvantage of this method which could be taken *self aldol condensation* between its own nucleophile. So, there are also researcher who like to react aldehyde first with base to reduce the possibilities of *self aldol condensation* [11]. From the study of its synthesis technique, in this research we also study the influence of reaction sequence to synthesis efficiency. The data of synthesis technique and solvent variation was listed in Table 2.

Table 2. Data of vanillin structure modification

No	Solvent	Synthesis sequence	Colour of resulting material	yield %
1	H ₂ O	NaOH, acetone, vanillin	Dark yellow	13.06
2	H ₂ O	NaOH, vanillin, acetone	Dark yellow	73.86
3	EtOH- H ₂ O 1:1	NaOH, acetone, vanillin	yellow	94.3
4	EtOH- H ₂ O 2:3	NaOH, vanillin, acetone	yellow	76.70
5	MeOH	NaOH, acetone, vanillin	Light yellow	90.2

The result of the synthesis using water have darker colour than ethanol-water and the lighter yellow was provided by methanol solvent. All resulting material was recrystallized using ethanol-water provided yellow gradated colour to dark brown for target compound number 1, 3, 4 and 5. It could be occurred due to the uncompleted reactions which reverse to result the reactant again, which confirm from TLC that showed those four materials were still vanillin. The compound no 2 was then analyzed by spectroscopy IR and NMR.

The FTIR data of vanillinacetone by structure modification showed sharp peak at 1620.21 cm⁻¹ (C-O carbonyl), shoulder on 3300 cm⁻¹ which indicated hydroxyl group. Methyl group was existed at 1327 cm⁻¹ and C-O-C appeared around 1000-1100 cm⁻¹. Characteristic absorption of aromatic appeared at 3055 cm⁻¹ was supported by sharp peak at 1573.91 cm⁻¹. C-H aldehyde characteristic at 2800 and 2700 cm⁻¹ was already disappearing. It could be hold up that the synthesis resulting vanillinacetone compound.

Data two dimensional of NMR spectrometer were needed to determine proton position at vanillin structure modification result and listed at Table 3 and the structure was shown in Figure 4.

Table 3. ¹H NMR, HMQC and HMBC spectral data of vanillinacetone

No C	$\delta H(\Sigma, m, J)$ ppm	δC ppm	HMBC ppm
1	2.2 (3; s)	27.28	197
2	-	197	
3	6.47 (1; d; 16)	124.8	
4	7.47 (1; d; 16)	144	124.8; 111.34; 197
2'	7.16 (1; s)	111.34	124.5; 144
3'	-	149.1	
3'-OCH ₃	3.9 (3; s)	56.29	149
5'	6.66 (1; d; 8.5)	116.4	124.5; 149
6'	7.04 (1; d; 8.5)	124.5	111.3; 144

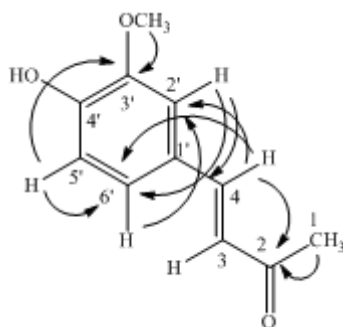


Figure 4. HMBC the modification of vanillinacetone

IR and NMR datas showed that the product of vanillin structure modification is vanillinacetone with dark yellow colour and give 73.86% in yield.

4. Conclusions

The conclusion of the research could be remark as:

- 4.1 Vanillin isolation with soxhlet extraction method using ethylacetate solvent resulted yield of 2.01 % with melting point of 81°C.
- 4.2 Vanillinacetone could be synthesized from isolated vanillin through its structure modification using crossed aldol condensation reaction to give yield of 73.86%.

5. Acknowledgments

The financial support from the Directorate General of Higher Education, Government of Indonesia, through the Project of Strategi Prioritas Nasional is gratefully acknowledged.

6. References

- [1]. Meynarti S.D.I., Laba U. dan Endang H., 2010, Balittri.litbang.deptan.go.id
- [2]. Unang Mansur, 2009, Teknik Penggunaan Naungan Paranet untuk Meningkatkan Pertumbuhan dan Produksi Vanili (*Vanilla planifoli* Andrews), *Buletin Teknik Pertanian*, Vol. 14, No. 2, 76-79.
- [3]. Cecep Virman, 2008, Teknik Inokulasi Mikoriza Arbuskula pada Bibit Vanili, *Buletin Teknik Pertanian*, Vol. 13 No. 2.
- [4]. Indriana, S.M., 2006, Ekstraksi Vanili Secara Enzimatis dari Buah Vanili (*Vanilla Planifolia* Andrews) Segar, Sekolah Pascasarjana, Bogor
- [5]. Dnyaneswar, J., Rekha B.N., Parag R. Gogate and Virendra K. Rathod, 2009, Extraction of Vanillin from Vanilla Pods : A Comparison Study of Conventional Soxhlet and Ultrasound Assisted Extraction, *Journal of Food Engineering*, Vol. 93, Issue 4, 421-426.
- [6]. V.E. Tarabanko, Yu.V. Chelbina, V.A. Sokolenko, N.V. Tarabanko, 2007, A Study of Vanillin Extraction by Octylamine, *Solvent Extraxtion and Ion Exchange*, Vol. 25, 99-107
- [7]. Sri Handayani dan Indyah, S.A., 2008, Synthesis of Hydroxyl Radical Scavengers from Benzalacetone and Its Derivatives, *Journal of Physical Chemistry*, Vol 19, No.2, 62-68.

- [8]. Sri Handayani, 2009, Synthesis and Activity Test of Two Asymmetric Dibenzalacetone as Potential Sunscreen Material, Proceeding of CBEE, Singapura
- [9]. Sri Handayani, Sabirin M., Chairil A. dan Sri Atun, 2010, Synthesis and Activity Test as Antioxidant of Two Hydroxydibenzalacetones, Proceeding of PACCON, Ubon Ratchatani, Thailand
- [10]. Goksu, S.; Celik, H.; Secen, H., 2003, An Efficient Synthesis of Alnustone, a Naturally Occuring Compound, *Turk. J. Chem.*, 27, 31-34.
- [11]. McBride, K., 2005, Synthesis of Dibenzalacetone by the Aldol Condensation, www.susuqu.edu, (24/1/2006)