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Report from The Organizing Committee

It is indeed my great pleasure and honor to welcome you all to Soedirman's International Conference on Mathematics and Applied Sciences (SICoMAS) 2019. The conference running this year is the first SICoMAS series hosted by Faculty of Mathematics and Natural Sciences Jenderal Soedirman University. As the development of technology and management of world resources for our future based on the innovation in Mathematics and Sciences, this conference takes issue "Innovation in Mathematics and Applied Sciences for better future".

SICoMAS 2019 aims to provide a platform for researchers, lecturers, teachers, students, practitioners, and industrial professionals to share knowledge, exchange ideas, collaborate, and present research results in the fields of Mathematics, Chemistry, Physics, and their applications. Hence, my sincere gratitude goes to our four keynote speakers (Prof. Dr. Hadi Nur from University Teknologi Malaysia, Prof. Dr. Hirokazu Saito from Tokyo University of Science, Dr. Devi Putra, ST, M.Sc. from Pertamina Research and Technology, and Uyi Sulaeman, Ph.D. from Jenderal Soedirman University), and our six invited speakers (Prof. Dr. Youtoh Imai from Nishogakusha University, Prof. Riyanto, Ph.D. from Universitas Islam Indonesia, Dr. Moh. Adhib Ulil Absor from Gadjah Mada University, Bambang Hendriya Guswanto, Ph.D, Dadan Hermawan, Ph.D. and Dr. Eng. Mukhtar Effendi, M. Eng. from Jenderal Soedirman University) for sharing their expertise in this conference. My deepest appreciation also goes to our 80 presenters and 7 non presenters for their commitment to participate in this conference.

As the output of this conference, some selected papers in the field of chemistry will be published in Jurnal Molekul which is accredited Sinta 1; and other selected papers in the fields of Mathematics, Physics, Physical Chemistry, and Innovative Chemistry Education will be published in IOP Conference Series Journal. So, I greatly thank Jenderal Soedirman University, all our contributors, and all the members of the committee for the invaluable support that makes this conference a reality.

Finally, I would like to apologize for any short comings found in this conference; and hopefully this two-day conference will be engraved in your memory.

The chair of SICoMAS 2019



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Table of contents

Volume 1494

2020

◀ Previous issue Next issue ▶

**Soedirman's International Conference on Mathematics and Applied Sciences (SICoMAS) 2019
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A Kamsyakawuni, M P Sari, A Riski and K A Santoso

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Model identification for prediction of dengue fever disease spreading using Bat Algorithm and backpropagation

A Damayanti, N L Hidayati and A B Pratiwi

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The proof of properties of dihedral group and its commutative elements



H Muhammad, L Susilowati and Fatmawati

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012004

Petri Net Model and Max-Plus Algebra on Queue in Clinic UNS Medical Center

T. Sulistyaningsih, Siswanto and Pangadi

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012005

Cat swarm optimization and Levenberg-Marquardt for model identification and prediction
identification and prediction tuberculosis disease spreading

A Nuari, A Damayanti and A B Pratiwi

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012006

Numerical comparisons based on new NCP-functions

I Nurhidayat and L C Han

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012007

Ant System Algorithm in Renewing Pheromone for Completing Quadratic Assignment
Problem

F M Utami, Renny and S R Nurshiami

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012008

Clustering of fish freshness using discrete wavelet transform and Kohonen self organizing
map

J Anvy, A Damayanti and A B Pratiwi

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012009

Annihilator graph of semiring of matrices over Boolean semiring

R Yudatama, V Y Kurniawan and S B Wiyono

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012010

Interval min-plus algebraic structure and matrices over interval min-plus algebra

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A R Awallia, Siswanto and V Y Kurniawan

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012011

On Cartesian product of intuitionistic Q-fuzzy ideal on ordered semigroup

N Hidayat, I Yanti, Z Fitriah, D Miftah, S Anam, S I Rahman and F Hidayat

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012012

Implementation of Bayesian Mixture Models in identifying subpopulation of breast cancer patients based on blood test measurements

N Dwimantara, S Abdullah, A Bustamam and A Rachman

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012013

Determining soil fertility using principal componen regression analysis of oil palm plantation in West Sulawesi, Indonesia

U S Pasaribu, N Nurhayati, N F F Ilmi and K N Sari

[+ Open abstract](#) [View article](#) [PDF](#)

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012014

Half-space model problem for a compressible fluid model of Korteweg type with slip boundary condition

Suma Inna, Sri Maryani and Hirokazu Saito

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012015

The metric dimension of k -subdivision graphs

L Susilowati, S Zahidah, R D Nastiti and M I Utoyo

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012016

Jacobson graph construction of ring \mathbb{Z}_3^n , for $n > 1$

A Novictor, L Susilowati and Fatmawati

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Betweenness Centrality in corona product of K_n and C_m graph

T I Haryadi and L Susilowati

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012018

Algorithm and computer program to determine metric dimension of graph

F Muhammad and L Susilowati

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[PDF](#)
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012019

An Adams type inequality of fractional integral operator on hypergroups

I Sihwaningrum, A Wardayani and Y Dasril

[+ Open abstract](#)
[View article](#)
[PDF](#)
OPEN ACCESS

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Regular rings and their properties

A Wardayani, I Kharismawati and I Sihwaningrum

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012021

The validity of the properties of real numbers set to hyperreal numbers Set

R Masithoh, B H Guswanto and S Maryani

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[View article](#)
[PDF](#)
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012022

Removal of rhodamine b using 4-hydroxy-3-methoxyphenylcalix[4]resorcinarene

S N Handayani, Irmanto and N N Indriyani

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012023

Adsorption of Gold(III), Copper(II), Nickel(II) on Amino Silica Hybrid Coated Magnetite

M T Thahir, N Nuryono and S J Santosa

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012024

Kinetics Adsorption of Strontium(II) by Silica Xerogel from Fly Ash

N A Kundari, K Megasari and C Yusephin

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[View article](#)
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012025

Triterpenoid and Steroid from the Rind of *Chisocheton macrophyllus* (Meliaceae)

V M Sari, Nurlelarsi, D Harneti, R maharani, N Indrayati, M N Azmi and U Supratman

[+ Open abstract](#)[View article](#)[PDF](#)

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012026

Introducing Colorimetric Analysis with Document Scanner for High School Students

A Fatoni, Supiani, D W Dwiasi and M D Anggraeni

[+ Open abstract](#)[View article](#)[PDF](#)

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012027

The pH dependence of Ag_3PO_4 synthesis on visible light photocatalytic activities

I Futihah, A Riapanitra, S Yin and U Sulaeman

[+ Open abstract](#)[View article](#)[PDF](#)

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012028

Improved reuse and affinity of enzyme using immobilized amylase on alginate matrix

Zusfahair, D R Ningsih, D Kartika, M Kurniasih, R Nofiani and A Fatoni

[+ Open abstract](#)[View article](#)[PDF](#)

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012029

Effect of ultraviolet and visible lights on degradation of congo red dye using $\text{Fe}^{2+}/\text{H}_2\text{O}_2$

J Tama, K Riyani and T Setyaningtyas

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012030

Introducing Iron Analysis with Smartphone Camera for High School Students

A Fatoni, Zusfahair and M D Anggraeni

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012031

Quantitative Structure Activity Relationship Analysis of 1,3,4-Thiadiazole Derivatives as Anti-Inflammatory using Parameterized Model 3 Method

Ponco Iswanto, Maylani Permata Saputri, Vaulina Y.D. Eva and Sari Paramita

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012032

Application of High Performance Liquid Chromatography Method for Triadimenol

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Analysis in Water Sample

D Hermawan, Cacu, Suwandri, A Fatoni, Mudasir and H Y Aboul-Enein

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012033

The Effect of Multiplanes Graphene Flakes Coating on Ag Nanowire Transparent Conductive

Nur Fiqalbi, Ratih Dwi Cahyaningrum, Bambang Prihandoko and Harsojo

[+ Open abstract](#)[View article](#)[PDF](#)**OPEN ACCESS**

012034

Mapping of gold mineralization using 3D inversion magnetic data at Zone X, West Java

Muzzammil Al Macky, Mohammad Syamsu Rosid and Agus Pajrin Jaman

[+ Open abstract](#)[View article](#)[PDF](#)**OPEN ACCESS**

012035

Hydrocarbon Reservoir Characterization of 'Y' Field in Kutai Basin East Kalimantan Using Artificial Neural Network Method

Y R Hendryan, M S Rosid and Haryono

[+ Open abstract](#)[View article](#)[PDF](#)**OPEN ACCESS**

012036

Study of Thin Film Cu Nanowire and Reduced Graphene Oxide as A Gas Sensor

Noviana Candra Wijayanti, Ratih Dwi Cahyaningrum and Harsojo

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012037

Study of Transparent Conductive of Ag Nanowire Under Ambient Environment

Ratih Dwi Cahyaningrum and Harsojo

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012038

Estimation of Potential of Iron Sand in The Eastern Coastal Area of Cilacap Regency Based on The Local Magnetic Anomalies Data

Sehah, M A Kurniawan, I Andriyanto, B Arismawan, A Risyad, A M Wibowo, S A Raharjo and Sugito

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Application of High Performance Liquid Chromatography Method for Triadimenol Analysis in Water Sample

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Application of High Performance Liquid Chromatography Method for Triadimenol Analysis in Water Sample

D Hermawan¹, Cacu¹, Suwandri¹, A Fatoni¹, Mudasir², and H Y Aboul-Enein³

¹Department of Chemistry, Faculty of Mathematics and Natural Science, Universitas Jenderal Soedirman (UNSOED), Purwokerto, Indonesia

²Department of Chemistry, Faculty of Mathematics and Natural Science, Universitas Gadjah Mada (UGM), Yogyakarta, Indonesia

³Department of Pharmaceutical and Medical Chemistry, Pharmaceutical and Drug Industries Research Division, National Research Centre (NRC), Dokki, Cairo, Egypt

Email: dadanphd@gmail.com

Abstract. Triadimenol is the most important triazole fungicide used for prevention and treatment of plant fungal disease. A simple high performance liquid chromatography (HPLC) method for triadimenol analysis in water sample is reported in this study. The optimized HPLC system using C₁₈ column was achieved using mobile phase composition containing acetonitrile-water (70:30, v/v), a flow rate of 1.0 mL/min, UV detection at 220 nm, and analysis time within 2.5 min. The calibration graph was linear in the range 5 to 50 mg/L with r^2 of 0.9997. The limit of detection (LOD) and limit of quantification (LOQ) obtained were 1.23 and 4.12 mg/L, respectively. Combination of solid phase extraction (SPE) and the optimized HPLC method has been successfully applied for the determination of triadimenol in water sample with a recovery of 94.24 % (RSD = 5.38%, $n = 4$). The proposed HPLC method provides short analysis time, high reproducibility and high sensitivity.

Keywords: Fungicides, HPLC, SPE, Triadimenol, Water sample.

1. Introduction

The major categories of pesticides classified by target are insecticides, herbicides, and fungicides. Fungicides are used to eradicate or prevent the undesirable growth of fungal microorganisms in many agricultural, horticultural, and industrial situations. Numerous substances possess antifungal activity, and their chemical structural spectrum is wide and diverse, covering both organic and inorganic substances. The most important category of fungicides is triazole-type because of their excellent protective and curative power against a wide spectrum of crop disease. Triadimenol {*1-(4-chlorophenoxy)-3,3-dimethyl-1-(1H-1,2,4-triazole-1-yl)butan-2-ol*}, one of the triazole fungicide, is a systemic fungicide for used protective, curative, and irritative. It is recommended for use as either fungicides or plant growth regulators [1-3].

Chromatographic methods used so far for the analysis of triadimenol include gas chromatography (GC) [4], supercritical fluid chromatography (SFC) [5], gel permeation chromatography and ultra performance convergence chromatography/tandem mass spectrometry [6]. High performance liquid chromatography (HPLC) method has also been used for triadimenol analysis. The advantages of HPLC method are rapid analysis, required small sample volume, column can be used many times, and high level of selectivity, sensitivity and accuracy [7-9].



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Combination of solid phase extraction (SPE) and the HPLC method has been successfully applied in this study for the determination of triadimenol in river water sample. The HPLC method using C₁₈ column and UV detection was optimized in this study. Structure of triadimenol is shown in figure 1.

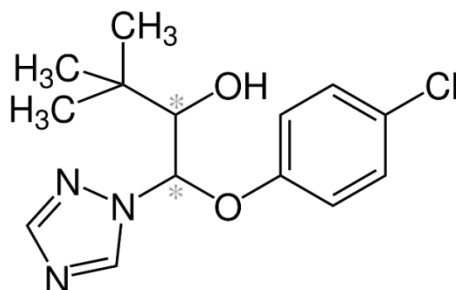


Figure 1. Chemical structure of triadimenol

2. Methods

2.1. Materials

Triadimenol was obtained from Riedel-de Haen (Seelze, Germany). All other chemicals and solvents were common brands of analytical reagent grade or better and were used as received. Deionized water was collected from a Millipore water purification system (Molsheim, France). All solvent were degassed prior to usage. River water sample was obtained from local river in Purwokerto, Indonesia. The stock solution of triadimenol (1000 mg/L) was prepared by dissolving triadimenol standard in methanol. A series of standard solutions (5 to 50 mg/L) were prepared by diluting the stock solution with methanol. The stock and all standard solutions were labeled and sealed with wrapping to avoid evaporation, and were stored in the refrigerator prior to use.

2.2. Instrumentation

The HPLC system with UV-Vis detection (Hitachi L-2000 series, Japan), equipped with a model L-2130 pump, an on-line solvent vacuum degasser, an autosampler with 10 μ L injector loop and a UV-Vis detector L-2420 at 220 nm. The separation was carried out in a Perkin Elmer C₁₈ μ m 150 x 4,6 mm. The mobile phase consisted of acetonitrile-water (v/v) and operated isocratically.

2.3. HPLC optimization

Triadimenol standard solution 50 mg/L was analyzed using the HPLC system with various of flow rates (0.8; 0.9; and 1.0 mL/min), and various of mobile phase acetonitrile-water compositions (70:30, 60:40, 50:50 v/v).

2.4. Solid phase extraction (SPE) procedure

A representative portion of water samples (river water sample and spiked river water sample containing triadimenol standard of 5 mg/L) were passed under vacuum through a C₁₈ solid-phase column (Supelclean SPE Tubes from Supelco), which was preconditioned with 5 mL of methanol and 5 mL of deionized water. Analytes were then eluted with 3 mL of methanol. The eluate was then diluted with methanol (total volume of 5 mL) before injected into the HPLC system,

2.5. Analytical performance of the HPLC method

The performance of the method was examined in terms of the linearity, repeatability, LOD and LOQ. Linearity of the HPLC method was assessed by constructing the calibration curves of peak areas against the concentration of standards (5 – 50 mg/L). The repeatability was determined in four replicates at triadimenol concentration of 50 mg/L. The limit of detection (LOD) and limit of

quantification (LOQ) were determined by the calibration curve along with the signal-to-noise ration (S/N) as 3 and 10, respectively.

3. Results and Discussion

3.1. Optimization of flow rate

The effect of different flow rate was first investigated in this study. Flow rate was varied in three different flow rates i.e. 0.8; 0.9; and 1.0 mL/min. The typical result of HPLC chromatogram of triadimenol with a different flow rate of the mobile phase is shown in figure 2. It can be seen that the retention time of triadimenol was gradually increased when the flow rate of mobile phase was decreased. The 1.0 mL/min has been chosen as the optimized flow rate because it gives the best baseline of triadimenol peak with retention time less than 2.5 min.

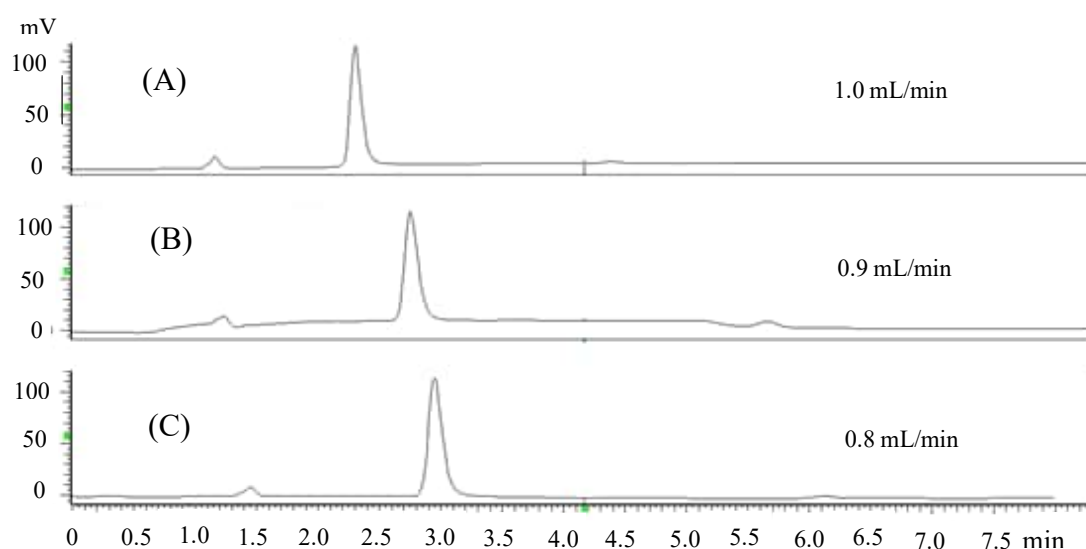


Figure 2. Chromatogram of triadimenol (50 mg/L in methanol) by HPLC with different flow rate conditions (A) 1.0 mL/min, (B) 0.9 mL/min, (C) 0.8 mL/min, mobile phase composition acetonitrile-water (60:40, v/v); injection volume 10 μ L.

3.2. Optimization of mobile phase

The effect of different mobile phase was then investigated in this study. The mobile phase varied was in three different percentages of acetonitrile-water (v/v) such as (50:50); (60:40); and (70:30). The typical result of the HPLC chromatogram of triadimenol with a different mobile phase is shown in figure 3. Addition of water reduce the elution strength of the mobile phase, therefore the analyte was eluted slower. The acetonitrile-water (70:30, v/v) has been chosen as the optimized mobile phase because it gives the best baseline of triadimenol peak with retention time less than 2.5 min.

3.3. Analytical performance of the HPLC method

The performance of the developed HPLC method was examined in terms of linearity, limit of detection (LOD), limit of quantification (LOQ) and repeatability. Calibration curve was linear over the

concentration range of 5 - 50 mg/L of the triadimenol standard with $r^2 = 0.9997$. The LOD and LOQ of triadimenol were 1.23 and 4.12 mg/L, respectively. The low LOD and LOQ showed that the developed HPLC method is sensitive and sufficient to determine triadimenol in water samples for routine analysis. The optimized HPLC method combined with solid phase extraction (SPE) pretreatment by using C_{18} solid-phase column is applicable for determination of triadimenol in the river water sample (figure 4). The percentage recovery obtained for analysis of triadimenol in water sample was 94.24 % with RSD of 5.41% ($n = 4$).

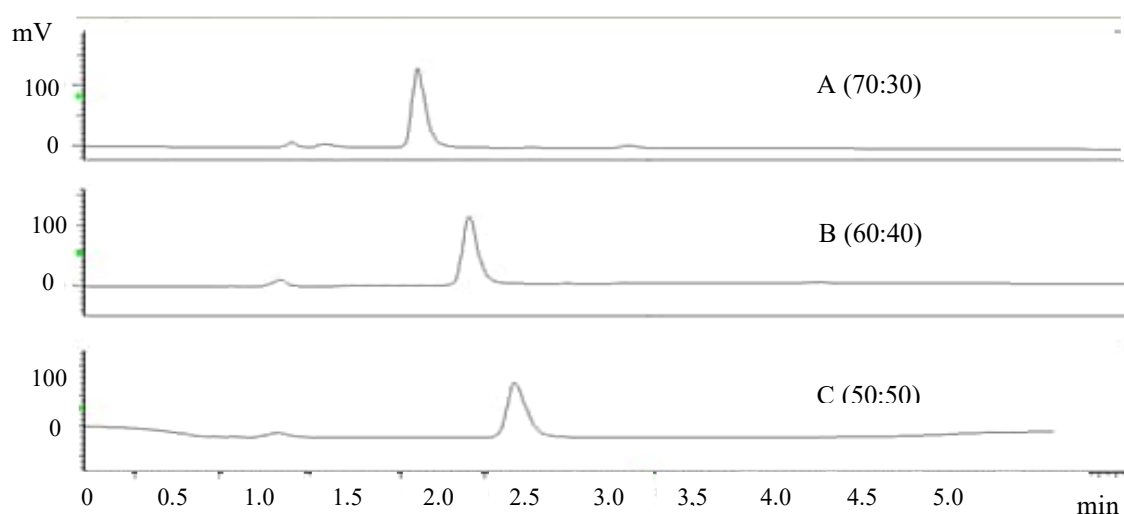


Figure 3. Chromatogram of triadimenol (50 mg/L in methanol) by HPLC with different mobile phase composition (A) acetonitrile-water (70:30, v/v), (B) acetonitrile-water (60:40, v/v), (C) acetonitrile-water (50:50, v/v); flow rate condition 1.0 mL/min; injection volume 10 μ L.

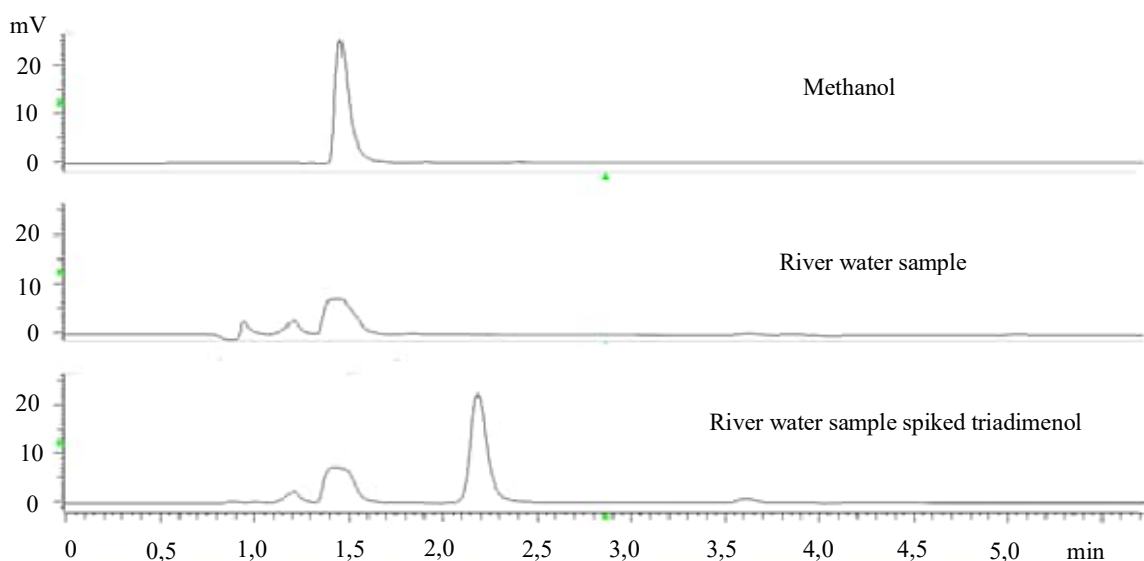


Figure 4. Chromatogram of solvent (methanol), river water sample and spiked water sample by triadimenol standard (5 mg/L), respectively by SPE-HPLC with UV detector (220 nm), mobile phase composition acetonitrile-water (70:30, v/v), flow rate condition 1.0 mL/min, injection volume 10 μ L.

4. Conclusions

In the present work, the high performance liquid chromatography (HPLC) method using C₁₈ column was successfully developed for the determination of triadimenol in the river water sample. The optimized HPLC condition was achieved using mobile phase composition containing acetonitrile-water (70:30, v/v), the flow rate of 1.0 mL/min, and UV detection at 220 nm. The calibration graph was linear in the range from 5 to 50 mg/L with r^2 of 0.9997. The limit of detection (LOD) and limit of quantification (LOQ) obtained were 1.23 and 4.12 mg/L, respectively. The optimized SPE-HPLC method was successfully applied to the analysis of triadimenol in the river water sample. The percentage recovery obtained for analysis of triadimenol in water sample was 94.24 % with RSD of 5.41% ($n = 4$).

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