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Selective Optical Chemosensors of Fe³⁺ Ions using 1*H*-Indole-2,3-dione

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Abstract: 1*H*-Indole-2,3-dione has attracted many attentions in the field of organic synthesis due to its easy modification with other organic compounds. On the other hand, 1*H*-indole-2,3-dione compound could also be used as chemosensor agent because it contains amide and carbonyl functional groups, which are able for binding as well as chelating metal ions. In the present work, we reported that 1*H*-indole-2,3-dione compound exhibits high sensing capability as well as high selective detection of Fe³⁺ ions through enhancement of its absorption peak at ultraviolet region. Particularly, a series of 0.5 mM of metal ions (Na⁺, K⁺, Ba²⁺, Ca²⁺, Co²⁺, Cu²⁺, Hg²⁺, Mg²⁺, Mn²⁺, Ni²⁺, Pb²⁺, Zn²⁺, Au³⁺, Al³⁺, Fe³⁺ and La³⁺) in distilled water was simply mixed with 0.05 mM of 1*H*-indole-2,3-dione in distilled water. Afterward, their UV-Vis spectral changes were recorded by using UV-Vis spectrophotometer. It was found that 1*H*-indole-2,3-dione itself gave two main absorption peaks at 242 and 302 nm. When the Fe³⁺ ions were added, the intensity of both absorption peaks was remarkably increased. From the UV-Vis titration experiment, limit of detection value was found to be 17.8 μM while limit of quantitation was found as 59.3 μM. This finding shows that the 1*H*-indole-2,3-dione gives a promising platform for fluorescent chemosensor agent for sensing of Fe³⁺ ion.

INTRODUCTION

Host-guest chemistry remains as an active research trends over the past several years [1-4]. Among them, nitrogen heterocyclic compounds have been extensively employed for their unique physicochemical properties [5]. Isatin or 1*H*-indole-2,3-dione was first obtained from the oxidation of indigo dye by acid oxidation such as nitric and chromic acids. The chemical structure of 1*H*-indole-2,3-dione is displayed in Figure 1. The 1*H*-indole-2,3-dione has been applied in many fields such as anticancer [6], antibacterial [7], corrosion [8], and chemosensor agent [9,10]. Furthermore, 1*H*-indole-2,3-dione has been utilized as raw material for organic synthesis because the phenyl ring, carbonyl and amide groups can be modified to form Schiff base, *N*-Mannich base, and other complex compounds [11-13].

Chemosensor from 1*H*-indole-2,3-dione derivatives as ligands, nanomaterials and nanocomposites were often reported due to their selectivity and sensitivity [14,15]. Besides that, as nanomaterial and nanocomposite, 1*H*-indole-2,3-dione derivatives have advantages such as high thermal stability, large pore size and high surface area [16,17].

Several researchers have used 1*H*-indole-2,3-dione derivatives for sensing Al³⁺ [16], Hg²⁺ [17], Fe³⁺ [18] and Cd²⁺ [19] ions. Dhara *et al.* in 2014 synthesized Schiff base derivative from 1*H*-indole-2,3-dione rhodamine scaffold and tested as a chemosensor to detect Al³⁺ ions [16]. This chemosensor showed high sensitivity in DMSO/H₂O (1:9 v/v) solvent, however several synthesis routes are necessary to synthesize 1*H*-indole-2,3-dione rhodamine scaffold. Lashgari *et al.* (2017) have reported that the grafting of commercial 1*H*-indole-2,3-dione with SBA-15 with (3-chloropropyl)trimethoxysilane as a linker and measured their emission with spectrofluorometer [17]. These nanocomposites were dispersed in water and used for sensing Hg²⁺ ions. It is well-known that the homogeneity from nanocomposite chemosensor is the main drawback for reproducible sensing method. Hence, it is necessary to develop a sensing method which employs a homogeneous system. Moreover, to the best of our knowledge, commercially available 1*H*-indole-2,3-dione as a chemosensor has not been evaluated previously for sensing metal ions. Whereas, 1*H*-indole-2,3-dione is promising to be applied as a chemosensor agent due to the presence of carbonyl and amide groups as well-known metal ion binding sites [20,21]. Therefore, in the present work, we reported the investigation of sensing Fe³⁺ ions in aqueous media using 1*H*-indole-2,3-dione as a chemosensor by evaluation of its selectivity and sensitivity.

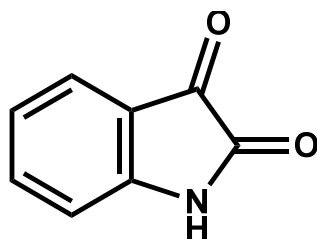


FIGURE 1. The structure of 1*H*-indole-2,3-dione

EXPERIMENTAL SECTION

General

1*H*-indole-2,3-dione (97%) and all other metals in chloride/nitrate salts were purchased from Sigma-Aldrich and were used as they are. The absorbance spectra were measured using spectrophotometer JASCO V-760 while the fluorescence spectra were measured using spectrofluorometer JASCO FP-8500ST. All measurements were performed using a quartz cuvette as a sample holder with 1 cm width.

Procedure

Optical Properties Study of 1H-Indole-2,3-dione

1*H*-indole-2,3-dione was dissolved in distilled water to obtain 0.1 mM concentration. After that, UV-Vis spectrum of 1*H*-indole-2,3-dione as the ligand were measured in the range of 200-800 nm. Fluorescent spectrum of this compound was particularly recorded using 3D measurement for each excitation-emission to the intensity in the range of 200-800 nm for every 10 nm.

The Spectroscopy Study of 1H-Indole-2,3-dione

Metal ions in chloride and nitrate salts were dissolved in distilled water for obtaining each metal ions stock solutions in 1 mM concentration. The spectroscopy study of 1*H*-indole-2,3-dione was carried out by contacting 1 mM of 1*H*-indole-2,3-dione (0.15 mL) with various of metal ion solution (0.15 mL) and distilled water to obtain the final volume of 3 mL. All absorbance spectra were recorded by using spectrophotometer UV-Vis.

Absorbance Spectral Titrations Study of 1H-Indole-2,3-dione

The UV-Vis titrations were conducted to calculate the limit of detection (LOD) and also limit of quantification (LOQ). The stock solution of 0.05 mM of the 1*H*-indole-2,3-dione was prepared by dissolving in distilled water.

Similarly, a 100 mM stock solution of Fe^{3+} was prepared separately in distilled water. The Fe^{3+} ions concentration was varied starting from 0.01, 0.025, 0.05, 0.075, .0.1, 0.25, 0.5 to 0.75 and 1 mM. Both solutions were mixed to obtain a solution with 3 mL as the total volume of the mixture, and then the changes in their UV-Vis spectra were recorded.

The Selectivity Study of 1H-Indole-2,3-dione

The selectivity study of 1*H*-indole-2,3-dione was performed to evaluate the capability of 1*H*-indole-2,3-dione to detect Fe^{3+} ions in the presence of other metal ions. The 0.5 mM of Fe^{3+} ions solution, 0.5 mM of other metal ions and 0.5 mM 1*H*-indole-2,3-dione solution in distilled water were mixed for this evaluation. The absorbance spectrum of the mixture was measured by UV-Vis spectrophotometer.

RESULTS AND DISCUSSION

Optical Properties Study

The photophysical properties of 1*H*-indole-2,3-dione were also systematically examined by using spectrofluorometer and spectrophotometer UV-Vis. The UV/visible absorption spectrum and 3D fluorescent spectrum were shown in Figure 2a-b. The UV-Vis spectrum in Figure 2a shows four absorption peaks at 208, 242, 302, and 418 nm. Absorption maxima at 208, 242, and 302 nm correspond to the $\pi \rightarrow \pi^*$ transition of the aromatic ring, $\pi \rightarrow \pi^*$ carbonyl C-2, and $\pi \rightarrow \pi^*$ carbonyl C-3, respectively. A weak absorption band at 418 nm correspond to the nitrogen and oxygen lone pair electrons ($n \rightarrow \pi^*$ transition) [22]. The 3D fluorescent spectrum of 1*H*-indole-2,3-dione in Figure 2b shows the blank area in the region 200-800 nm for the emission and excitation wavelength. This result indicates that 1*H*-indole-2,3-dione in distilled water does not have fluorescence phenomena due to the formation of aggregation (Aggregation-caused Quenching) [23]. The aggregation caused by overlaps in two π -conjugated systems (π - π stacking) between benzene ring from two 1*H*-indole-2,3-dione molecules. From the aforementioned above, in this work, the sensing method of 1*H*-indole-2,3-dione was evaluated through UV-Vis spectrometry method.

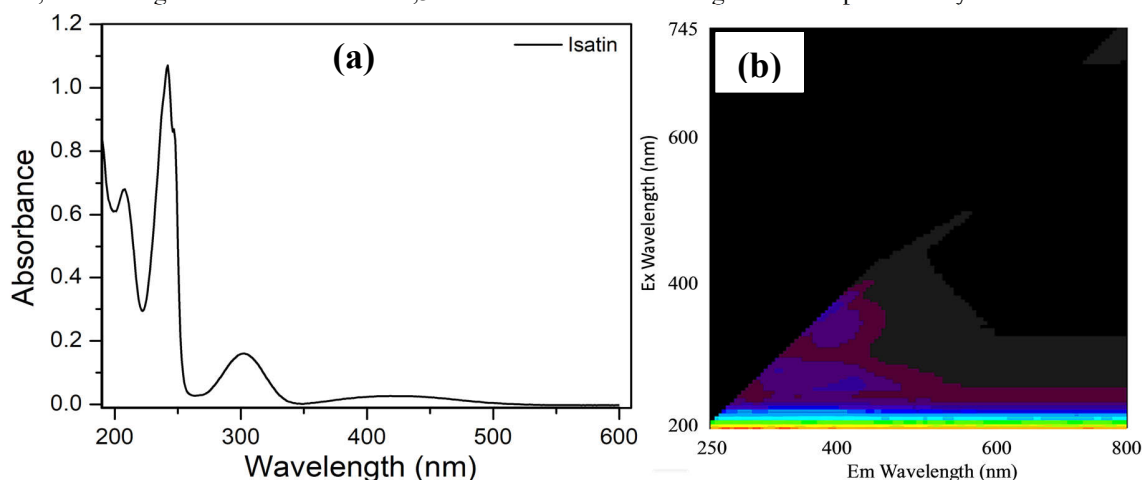


FIGURE 2. (a) The UV-Vis absorption and (b) the fluorescent 3D spectra of 1*H*-indole-2,3-dione 0.05 mM

The Spectroscopy Studies

The spectroscopy studies were used to evaluate the sensing capability of 1*H*-indole-2,3-dione as a chemosensor. Particularly, the selectivity profile of 1*H*-indole-2,3-dione sensor against different metal ions were also investigated. The UV-Vis spectral changes of the 1*H*-indole-2,3-dione solution after addition of various metal ions solution, such as Na^+ , K^+ , Ba^{2+} , Ca^{2+} , Co^{2+} , Cu^{2+} , Hg^{2+} , Mg^{2+} , Mn^{2+} , Ni^{2+} , Pb^{2+} , Zn^{2+} , Au^{3+} , Al^{3+} , Fe^{3+} and La^{3+} show in Figure 3a. As shown in the Figure 3a, it is interesting to note that only Fe^{3+} ions were able to induce strong absorption peaks at 242 and 302 nm due to complexation phenomenon between 1*H*-indole-2,3-dione at carbonyl group with Fe^{3+} ions.

After the addition of metal ions, the color of the solution was changed from pale-yellow to bright yellow as shown in Figure 3b. Hence, the changes in UV-Vis spectrum of 1*H*-indole-2,3-dione solution for sensing Fe³⁺ ions were well supported by its color as bright yellow.

Titration Study

The UV-Vis titration spectral changes of 1*H*-indole-2,3-dione in the presence of different concentrations of Fe³⁺ ions were recorded to calculate the LOD and LOQ [24]. As showed in Figure 4a, it was clearly showed that the addition of Fe³⁺ ions with higher concentration (0.01-1 mM) gave a gradual increment of the absorption intensity at 242 and 302 nm. Thus, from the calibration curve, a linear correlation between the concentration of Fe³⁺ and the absorption intensity was achieved as shown in Figure 4b. The linear equation of $dI/I_0 = 8.9643 [Fe^{3+}] + 0.132$ was obtained with 0.9985 as the correlation coefficient. It was found that the LOD and LOQ values were 17.8 and 59.3 μ M, demonstrating that 1*H*-indole-2,3-dione is a potential chemosensor agent to detect Fe³⁺ ions even at lower concentration (good sensitivity). Compared to other previous results, the LOD and LOQ of 1*H*-indole-2,3-dione is the best as shown in Table 1.

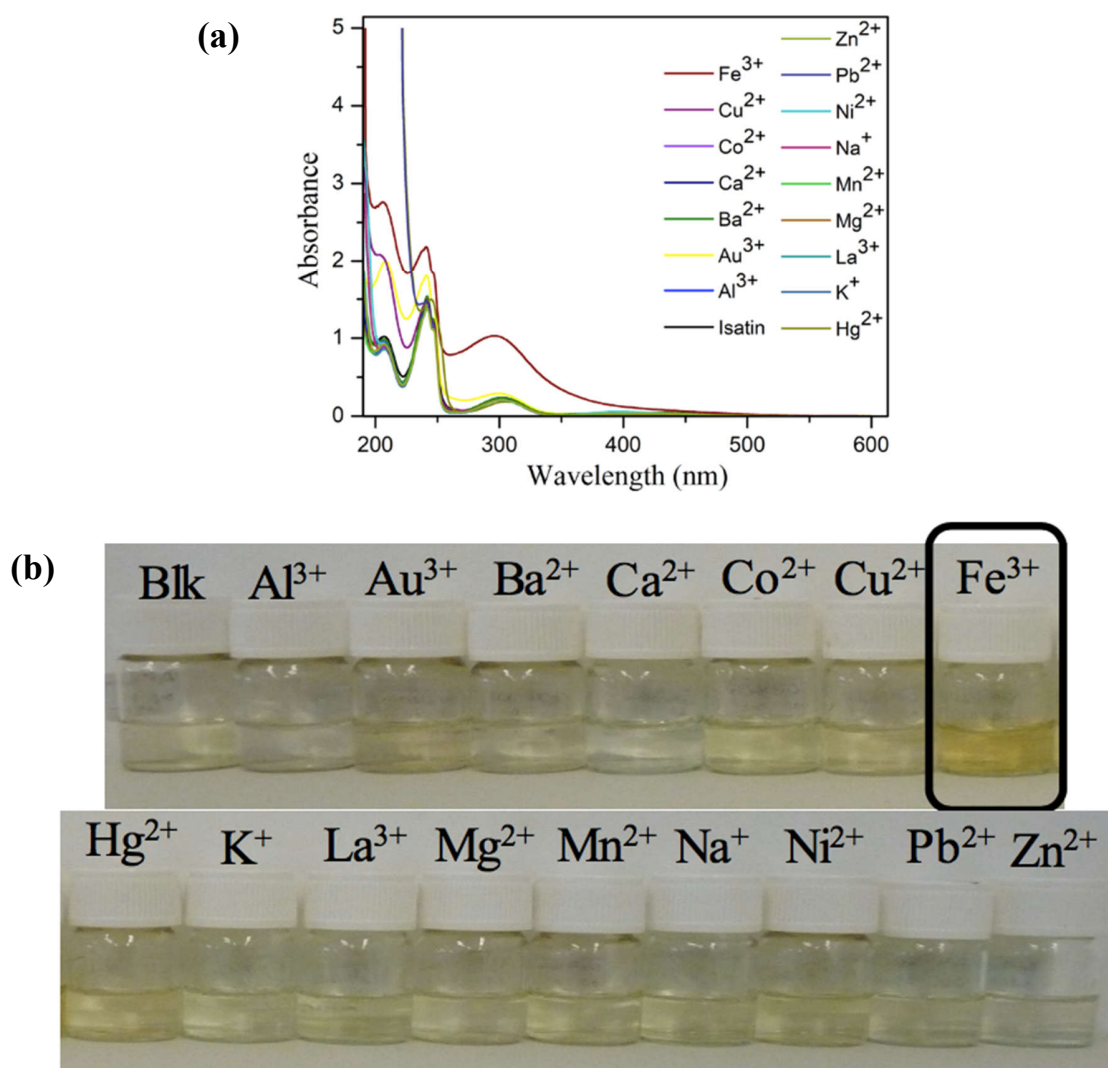


FIGURE 3. (a) The UV-Vis absorption spectral changes of 1*H*-indole-2,3-dione (0.05 mM) in distilled water in response to various metal ions with concentration of 0.5 mM and (b) their color changes upon addition of 10 equiv. of different metal

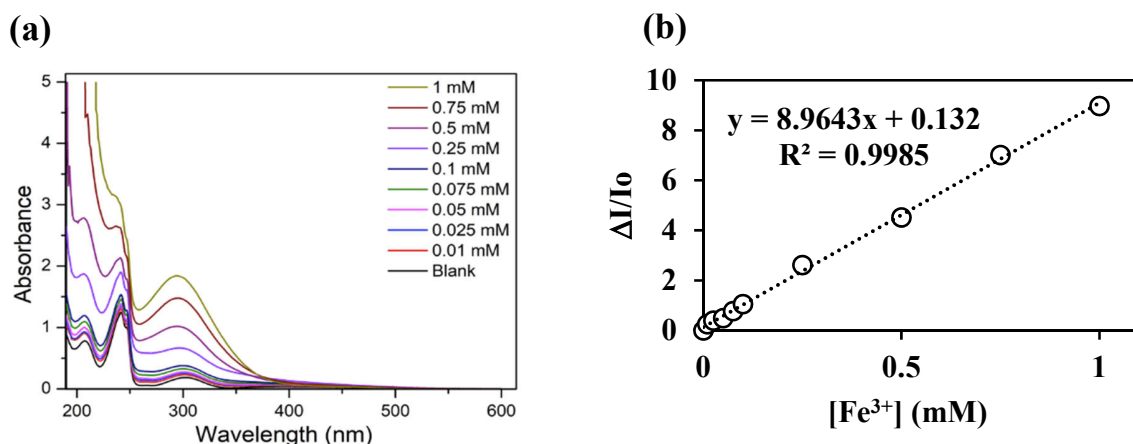


FIGURE 4. (a) The UV-vis absorption spectral changes of *1H*-indole-2,3-dione (0.05 mM) in distilled water upon gradual addition of Fe^{3+} ion with concentration 0.01, 0.025, 0.05, 0.075, 0.1, 0.25, 0.5, 0.75 and 1 mM and (b) the calibration curve plot of *1H*-indole-2,3-dione by Fe^{3+} ions presented in the form of $(\Delta I/I_0)$ vs $[\text{Fe}^{3+}]$

The Selectivity Study

The sensing capability of *1H*-indole-2,3-dione toward Fe^{3+} ions in the presence of other competing metal ions is pivotal parameter to evaluate its performance. Therefore, the interference study was critical to be investigated by addition of Fe^{3+} ions with the presence of other metal ions into a *1H*-indole-2,3-dione solution. The absorption spectral changes of the mixture were recorded and shown as Figure 5 in its bar chart. As shown in Figure 5, the presence of other metal ions did not show remarkable interference to the absorption of change of *1H*-indole-2,3-dione toward Fe^{3+} ions. These results demonstrate that *1H*-indole-2,3-dione as a chemosensor is highly selective to Fe^{3+} ions even with the involving of interferences from other metal ions.

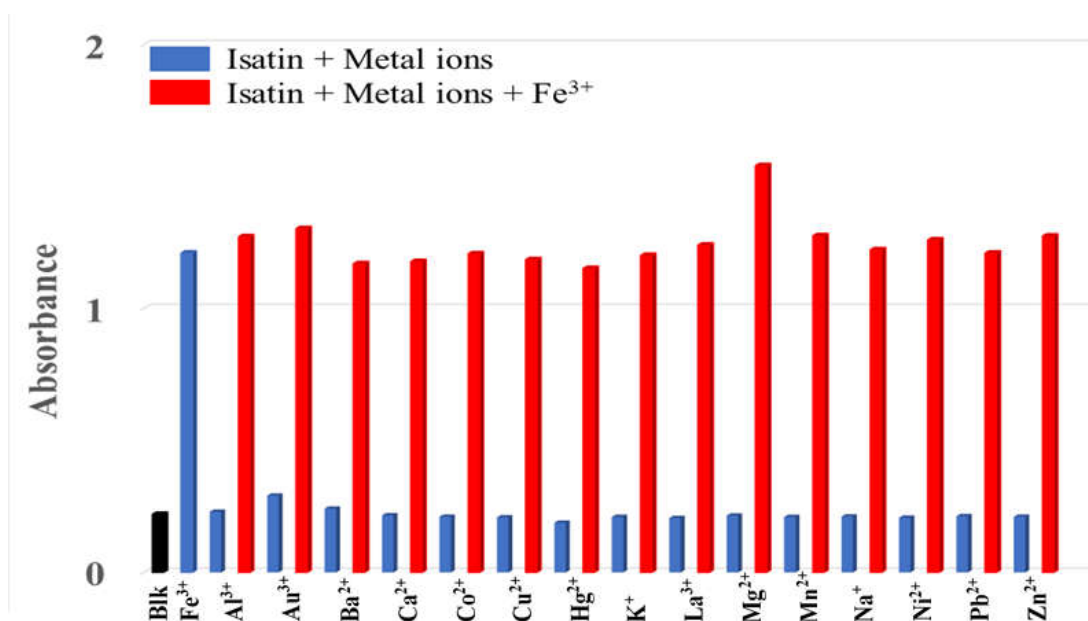
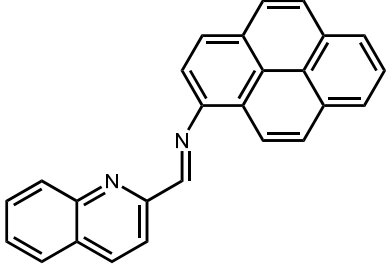
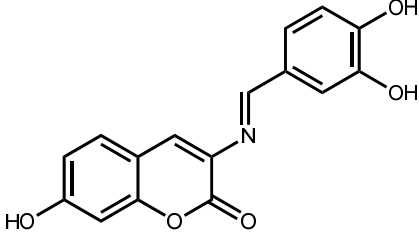
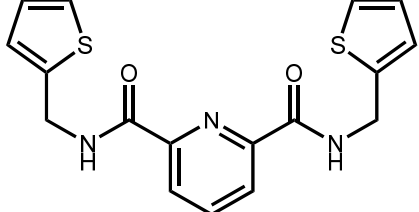
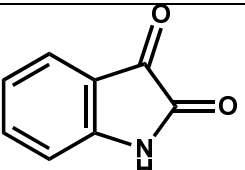


FIGURE 5. The competitive study of isatin with Fe^{3+} and the presence of other metal ions. black bar: absorption intensity of blank (*1H*-indole-2,3-dione 0.05 mM) in distilled water. Blue bar: absorption intensity of *1H*-indole-2,3-dione (0.05 mM) with other metal ions (0.5 mM) in distilled water. Red bar: absorption intensity of *1H*-indole-2,3-dione (0.05 mM) with Fe^{3+} ions (0.5 mM) and the addition of the respective competing cations (0.5 mM) in distilled water

TABLE 1. The comparison of the analytical performance between isatin-Fe³⁺ with other recently reported sensors

Chemosensor	Limit of Detection	Testing Media	Reff.
	32.2 μM	DMSO/H ₂ O (v/v=70:30)	[25]
	51.7 μM	HEPES buffer, pH 7.4, 1% DMSO	[26]
	8760 μM	MeOH	[27]
	17.0 μM	H ₂ O	Present work

CONCLUSION

In conclusion, 1*H*-indole-2,3-dione is potential to be used as a selective chemosensor for Fe³⁺ ions by utilizing a UV-Vis spectrophotometer for the evaluation of its absorption spectral changes. Among all evaluated monovalent, divalent and trivalent metal ions (Na⁺, K⁺, Ba²⁺, Ca²⁺, Co²⁺, Cu²⁺, Hg²⁺, Mg²⁺, Mn²⁺, Ni²⁺, Pb²⁺, Zn²⁺, Au³⁺, Al³⁺, Fe³⁺ and La³⁺), the a complex between 1*H*-indole-2,3-dione and Fe³⁺ ions could selectively increase the absorption band of the ligand at 242 and 302 nm at carbonyl absorption band. This interaction provides limit of detection in 17.8 μM and limit of quantification in 59.3 μM that are more sensitive than previously reported ones. Moreover, the solution appearance of the complex between 1*H*-indole-2,3-dione and Fe³⁺ ion gave a bright yellow color to support the presence of the binding which was clearly visible to the naked eye for direct recognition. Such interaction does not significantly influence by the presence of other metal ions as interferences, which is remarkable for the development of chemosensors. Hence, these findings are pivotal for further investigation on design a selective chemosensor based on nitrogen heterocyclic moieties.

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- 2235 (2020) ▾
- 2233 (2020) ▾
- 2230 (2020) ▾
- 2220 (2020) ▾
- 2232 (2020) ▾
- 2231 (2020) ▾
- 2229 (2020) ▾
- 2228 (2020) ▾
- 2226 (2020) ▾
- 2222 (2020) ▾
- 2217 (2020) ▾
- 2223 (2020) ▾
- 2216 (2020) ▾
- 2215 (2020) ▾
- 2221 (2020) ▾
- 2211 (2020) ▾
- 2225 (2020) ▾
- 2213 (2020) ▾
- 2209 (2020) ▾
- 2218 (2020) ▾
- 2214 (2020) ▾
- 2212 (2020) ▾
- 2207 (2020) ▾
- 2210 (2020) ▾
- 2208 (2020) ▾
- 2206 (2020) ▾

Table of Contents

< PREV NEXT >

PROCEEDINGS OF THE 5TH INTERNATIONAL SYMPOSIUM ON APPLIED CHEMISTRY 2019



Conference date: 23–24 October 2019
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PRELIMINARY

🔒 No Access . November 2019

Preface: Proceedings of the 5th International Symposium on Applied Chemistry 2019

AIP Conference Proceedings 2175, 010001 (2019);
<https://doi.org/10.1063/1.5134563>

🔒 No Access . November 2019

Scientific Committee: Proceedings of the 5th International Symposium on Applied Chemistry 2019

AIP Conference Proceedings 2175, 010002 (2019);
<https://doi.org/10.1063/1.5134564>

ARTICLES

🔒 No Access . November 2019

Baking quality, texture and sensory evaluation of gluten free cake made from modified taro flour

Riyanti Ekafitri, Yudi Pranoto and Ainia Herminiati

AIP Conference Proceedings 2175, 020001 (2019);
<https://doi.org/10.1063/1.5134565>

SHOW ABSTRACT

🔒 No Access . November 2019


Latex-starch hybrid synthesis using CGDE method with ethanol addition and air injection

Anisa Uswatun Hasanah, Adream Bais Junior and Nelson Saksono

AIP Conference Proceedings 2175, 020002 (2019);
<https://doi.org/10.1063/1.5134566>

- 2205 (2020) ∨
- 2204 (2020) ∨
- 2203 (2020) ∨
- 2197 (2020) ∨
- 2202 (2019) ∨
- 2182 (2019) ∨
- 2199 (2019) ∨
- 2198 (2019) ∨
- 2200 (2019) ∨
- 2192 (2019) ∨
- 2194 (2019) ∨
- 2201 (2019) ∨
- 2195 (2019) ∨
- 2191 (2019) ∨
- 2188 (2019) ∨
- 2196 (2019) ∨
- 2190 (2019) ∨
- 2193 (2019) ∨
- 2187 (2019) ∨
- 2186 (2019) ∨
- 2180 (2019) ∨
- 2185 (2019) ∨
- 2183 (2019) ∨
- 2174 (2019) ∨
- 2184 (2019) ∨
- 2177 (2019) ∨
- 2179 (2019) ∨
- 2178 (2019) ∨
- 2189 (2019) ∨
- 2181 (2019) ∨
- 2176 (2019) ∨
- 2175 (2019) ∧
- Issue 1, November 19
- 2167 (2019) ∨
- 2171 (2019) ∨
- 2172 (2019) ∨
- 2173 (2019) ∨
- 2169 (2019) ∨
- 2170 (2019) ∨

SHOW ABSTRACT


 No Access . November 2019

Comparing nitrate production through CGDE (contact glow discharge electrolysis) using Na₂SO₄ solution and K₂SO₄ solution

Puteri Salsabila, Ardiansah Haryansyah and Nelson Saksono

AIP Conference Proceedings 2175, 020003 (2019);
<https://doi.org/10.1063/1.5134567>

SHOW ABSTRACT


 No Access . November 2019

Performance of phenolic wastewater degradation with ozonation and catalytic ozonation technique in multi injection bubble column reactor

Sarah Vania Chaisani, Dionisius Parsaoran Wahyudi, Enjarlis, Eva Fathul Karamah and Setjo Bismo

AIP Conference Proceedings 2175, 020004 (2019);
<https://doi.org/10.1063/1.5134568>

SHOW ABSTRACT


 No Access . November 2019

Removal of organic and inorganic (phenolic and iron compound) pollutants from wastewater using DBD cold plasma reactor

Ratu Anissa Cahyani, Yulia Endah Permata, Eva Fathul Karamah and Setjo Bismo

AIP Conference Proceedings 2175, 020005 (2019);
<https://doi.org/10.1063/1.5134569>

SHOW ABSTRACT


 No Access . November 2019

Biodiesel synthesis in DBD plasma reactor using hot mixture of castor oil and used palm oil - Methanol

Kania Zara, Sesia Fitri Anisa, Shafira Nabilla and Setjo Bismo

AIP Conference Proceedings 2175, 020006 (2019);
<https://doi.org/10.1063/1.5134570>

SHOW ABSTRACT


 No Access . November 2019

Application of edible film from heat-moisture treated sweet potato starch on the quality of pineapple dodol

Novita Indrianti and Lia Ratnawati

AIP Conference Proceedings 2175, 020007 (2019);
<https://doi.org/10.1063/1.5134571>

SHOW ABSTRACT

 No Access . November 2019


Physicochemical, structural and morphological properties of some arrowroot (*Maranta arundinacea*) accessions growth in Indonesia

- 2168 (2019) ▼
- 2162 (2019) ▼
- 2166 (2019) ▼
- 2165 (2019) ▼
- 2164 (2019) ▼
- 2163 (2019) ▼
- 2161 (2019) ▼
- 2160 (2019) ▼
- 2159 (2019) ▼
- 2158 (2019) ▼
- 2157 (2019) ▼
- 2156 (2019) ▼
- 2153 (2019) ▼
- 2155 (2019) ▼
- 2154 (2019) ▼
- 2152 (2019) ▼
- 2150 (2019) ▼
- 2148 (2019) ▼
- 2151 (2019) ▼
- 2142 (2019) ▼
- 2141 (2019) ▼
- 2147 (2019) ▼
- 2145 (2019) ▼
- 2149 (2019) ▼
- 2139 (2019) ▼
- 2144 (2019) ▼
- 2138 (2019) ▼
- 2146 (2019) ▼
- 2143 (2019) ▼
- 2140 (2019) ▼
- 2136 (2019) ▼
- 2135 (2019) ▼
- 2137 (2019) ▼
- 2134 (2019) ▼
- 2132 (2019) ▼
- 2129 (2019) ▼
- 2131 (2019) ▼
- 2125 (2019) ▼
- 2133 (2019) ▼

Enny Sholichah, Puspita Deswina, Achmat Sarifudin, Cecep Erwan
Andriansyah and Nurhaedar Rahman

AIP Conference Proceedings 2175, 020008 (2019);
<https://doi.org/10.1063/1.5134572>

SHOW ABSTRACT


 No Access . November 2019

Physicochemical, baking quality, and sensory evaluation of gluten free bread made from modified sweet potato flour with addition of nuts flour

Dewi Desnilasari, Nok Afifah and Novita Indrianti

AIP Conference Proceedings 2175, 020009 (2019);
<https://doi.org/10.1063/1.5134573>

SHOW ABSTRACT

 No Access . November 2019

Effect of the acid-base properties of the support on the performance of ruthenium catalysts in the hydrodeoxygenation of stearic acid

Isni Putri Setyoningsih, Nino Rinaldi, Isalmi Aziz, Muhammad Ridwan, Sudyarmanto, Yati Maryati, Adid Adep Dwiatmoko and Fauzan Aulia

AIP Conference Proceedings 2175, 020010 (2019);
<https://doi.org/10.1063/1.5134574>

SHOW ABSTRACT


 No Access . November 2019

Effect of antimicrobials addition on the characteristic of arrowroot starch-based films

Lia Ratnawati and Nok Afifah

AIP Conference Proceedings 2175, 020011 (2019);
<https://doi.org/10.1063/1.5134575>

SHOW ABSTRACT


 No Access . November 2019

Biocomposite of pectin and starch filled with nanocrystalline cellulose (NCC): The effect of filler loading and glycerol addition

M. Thoriq Al Fath, Halimatuddahlilana Nasution, Hamidah Harahap and Chendis Ekawati Ayu

AIP Conference Proceedings 2175, 020012 (2019);
<https://doi.org/10.1063/1.5134576>

SHOW ABSTRACT


 No Access . November 2019

Formulation of herbal tea drinks by adding green tea to improve antioxidant activities

Megawati, Teni Ernawati, Lia Meilawati, Indah D. Dewijanti and Edi Supriadi

AIP Conference Proceedings 2175, 020013 (2019);
<https://doi.org/10.1063/1.5134577>

SHOW ABSTRACT

 No Access . November 2019


2130 (2019) ✓
2126 (2019) ✓
2124 (2019) ✓
2116 (2019) ✓
2128 (2019) ✓
2121 (2019) ✓
2127 (2019) ✓
2123 (2019) ✓
2122 (2019) ✓
2119 (2019) ✓
2115 (2019) ✓
2120 (2019) ✓
2113 (2019) ✓
2117 (2019) ✓
2118 (2019) ✓
2111 (2019) ✓
2114 (2019) ✓
2112 (2019) ✓
2109 (2019) ✓
2110 (2019) ✓
2108 (2019) ✓
2107 (2019) ✓
2106 (2019) ✓
2105 (2019) ✓
2102 (2019) ✓
2104 (2019) ✓
2103 (2019) ✓
2100 (2019) ✓
2097 (2019) ✓
2098 (2019) ✓
2101 (2019) ✓
2094 (2019) ✓
2093 (2019) ✓
2090 (2019) ✓
2099 (2019) ✓
2096 (2019) ✓
2095 (2019) ✓
2092 (2019) ✓
2091 (2019) ✓

Formulation of instant porridge by using natural folic acid fortificant as a complementary infant feeding

Agustine Susilowati, Yati Maryati and Aspiyanto

AIP Conference Proceedings 2175, 020014 (2019);
<https://doi.org/10.1063/1.5134578>

SHOW ABSTRACT


 No Access . November 2019

Synthesis N-palmitoyl lysine from palmitate acid and L-lysine used mixed solvent: Effect of temperature and reaction time

M. Syukri, N. R. Purba, B. R. Hutajulu, D. Alfizah, A. Hutagalung and Z. Masyithah

AIP Conference Proceedings 2175, 020015 (2019);
<https://doi.org/10.1063/1.5134579>

SHOW ABSTRACT


 No Access . November 2019

Recovering organic acids fermented beetroot (*Beta vulgaris* L.) through microfiltration to prevent increase of natural cholesterol

Agustine Susilowati, Aspiyanto, Puspa D. Lotulung, Yati Maryati and Hani Mulyani

AIP Conference Proceedings 2175, 020016 (2019);
<https://doi.org/10.1063/1.5134580>

SHOW ABSTRACT


 No Access . November 2019

The performance of microfiltration membrane of total polyphenol from fermented beetroot (*Beta vulgaris* L.) to prevent natural oxidation

Aspiyanto, Agustine Susilowati, Puspa D. Lotulung, Yati Maryati and Hani Mulyani

AIP Conference Proceedings 2175, 020017 (2019);
<https://doi.org/10.1063/1.5134581>

SHOW ABSTRACT


 No Access . November 2019

Evaluation of antioxidant activity of formulated functional drinks derived from katuk (*Sauropus androgynous*) leaf extracts: Optimization using response surface methodology (RSM)

Yati Maryati, Agustine Susilowati, Aspiyanto, Hani Mulyani, Nina Artanti and Setyani Budiari

AIP Conference Proceedings 2175, 020018 (2019);
<https://doi.org/10.1063/1.5134582>

SHOW ABSTRACT

 No Access . November 2019


Characterizations of activated zeolite using hydrolysis method

Halimatuddahlana Nasution, Hamidah Harahap, Setiaty Pandia, Danil Maha Putra and M. Thoriq Al Fath

- 2089 (2019) ✓
- 2086 (2019) ✓
- 2088 (2019) ✓
- 2084 (2019) ✓
- 2082 (2019) ✓
- 2078 (2019) ✓
- 2085 (2019) ✓
- 2083 (2019) ✓
- 2087 (2019) ✓
- 2080 (2019) ✓
- 2081 (2019) ✓
- 2079 (2019) ✓
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- 2055 (2019) ✓
- 2064 (2019) ✓
- 2054 (2019) ✓
- 2063 (2019) ✓
- 2059 (2019) ✓
- 2057 (2019) ✓
- 2061 (2019) ✓
- 2058 (2019) ✓
- 2052 (2018) ✓
- 2056 (2018) ✓
- 2050 (2018) ✓
- 2053 (2018) ✓

AIP Conference Proceedings 2175, 020019 (2019);
<https://doi.org/10.1063/1.5134583>

SHOW ABSTRACT


 No Access . November 2019

Application of graphite furnace atomic absorption spectroscopy (GF-AAS) on determination of lead in bay (*Syzygium polyanthum*) leaves

Zatil Afrah Athaillah, Latifahatul Husna, Nurlathifah and Indah Dwiatmi Dewijanti

AIP Conference Proceedings 2175, 020020 (2019);
<https://doi.org/10.1063/1.5134584>

SHOW ABSTRACT


 No Access . November 2019

Antimicrobial activity of bay leaf (*Syzygium polyanthum* (wight) walp) extracted using various solvent

Indah Dwiatmi Dewijanti, Wibowo Mangunwardoyo, Astari Dwianti, Muhammad Hanafi, Nina Artanti, Tjandrawati Mozef and Anastasia Fitria Devi

AIP Conference Proceedings 2175, 020021 (2019);
<https://doi.org/10.1063/1.5134585>

SHOW ABSTRACT


 No Access . November 2019

Semi chemically-processed nano fiber cellulose isolated from palm fiber waste: Morphology and physical characterization

Nuim Hayat, Hamidah Harahap and Halimatuddahlia Nasution

AIP Conference Proceedings 2175, 020022 (2019);
<https://doi.org/10.1063/1.5134586>

SHOW ABSTRACT


 No Access . November 2019

Antidiabetic and toxicity activities of *Cinchona ledgeriana* leaves extracts

Andini Sundowo, Minarti and Galuh Widiyarti

AIP Conference Proceedings 2175, 020023 (2019);
<https://doi.org/10.1063/1.5134587>

SHOW ABSTRACT


 No Access . November 2019

The effect of lactic acid fermentation in antioxidant activity and total polyphenol contents of the banana (*Musa acuminata Linn*) juice

Setyani Budiari, Yati Maryati, Agustine Susilowati, Hani Mulyani and Puspa Dewi Narij Lotulung

AIP Conference Proceedings 2175, 020024 (2019);
<https://doi.org/10.1063/1.5134588>

SHOW ABSTRACT

 No Access . November 2019

- 2049 (2018) ✓
- 2051 (2018) ✓
- 2048 (2018) ✓
- 2045 (2018) ✓
- 2046 (2018) ✓
- 2040 (2018) ✓
- 2031 (2018) ✓
- 2047 (2018) ✓
- 2039 (2018) ✓
- 2043 (2018) ✓
- 2044 (2018) ✓
- 2037 (2018) ✓
- 2041 (2018) ✓
- 2038 (2018) ✓
- 2035 (2018) ✓
- 2036 (2018) ✓
- 2030 (2018) ✓
- 2042 (2018) ✓
- 2033 (2018) ✓
- 2024 (2018) ✓
- 2022 (2018) ✓
- 2028 (2018) ✓
- 2027 (2018) ✓
- 2034 (2018) ✓
- 2029 (2018) ✓
- 2026 (2018) ✓
- 2025 (2018) ✓
- 2032 (2018) ✓
- 2023 (2018) ✓
- 2021 (2018) ✓
- 2019 (2018) ✓
- 2020 (2018) ✓
- 2013 (2018) ✓
- 2017 (2018) ✓
- 2018 (2018) ✓
- 2016 (2018) ✓
- 2015 (2018) ✓
- 2014 (2018) ✓
- 2011 (2018) ✓

Benefits of fermented beet (*Beta vulgaris* L.) against digestive infection *Escherichia coli* and free radicals prevention

Hani Mulyani, Yati Maryati, Euis Filaila, Agustine Susilowati, Puspa D. N. Lotulung and Aspiyanto Aspiyanto

AIP Conference Proceedings 2175, 020025 (2019);
<https://doi.org/10.1063/1.5134589>

SHOW ABSTRACT

No Access . November 2019

Fabrication of zinc oxide nanostructure as antibacterial agent

Ananda Miftahul Husna and Fransiska Sri Herwahyu Krismastuti

AIP Conference Proceedings 2175, 020026 (2019);
<https://doi.org/10.1063/1.5134590>

SHOW ABSTRACT

No Access . November 2019

Preparation of activated carbon by chemical activation using KOH and acetone from low density polyethylene (LDPE) wastes

Yuliusman Yuliusman, Mega Puspitasari and Azmia Rizka Nafisah

AIP Conference Proceedings 2175, 020027 (2019);
<https://doi.org/10.1063/1.5134591>

SHOW ABSTRACT

No Access . November 2019

Modification of low-density polyethylene based activated carbon using titanium dioxide for carbon monoxide and hydrocarbon adsorption

Yuliusman, Mega Puspita Sari and Azmia Rizka Nafisah

AIP Conference Proceedings 2175, 020028 (2019);
<https://doi.org/10.1063/1.5134592>

SHOW ABSTRACT

No Access . November 2019

Secondary metabolite compound isolated from the leaves of *Macaranga magna* Turrill

Minarti, Antonius Herry Cahyana and Akhmad Darmawan

AIP Conference Proceedings 2175, 020029 (2019);
<https://doi.org/10.1063/1.5134593>

SHOW ABSTRACT

No Access . November 2019

The effect of drying methods on chemical and physical properties of leaves and stems *Moringa oleifera* Lam

Woro Setiaboma, Dita Kristanti and Ainia Herminiati

AIP Conference Proceedings 2175, 020030 (2019);
<https://doi.org/10.1063/1.5134594>

SHOW ABSTRACT

No Access . November 2019


- 2012 (2018) ✓
- 2010 (2018) ✓
- 2008 (2018) ✓
- 2009 (2018) ✓
- 2007 (2018) ✓
- 2006 (2018) ✓
- 2004 (2018) ✓
- 2005 (2018) ✓
- 2001 (2018) ✓
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- 1982 (2018) ✓
- 1995 (2018) ✓
- 1984 (2018) ✓
- 1986 (2018) ✓
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- 1987 (2018) ✓
- 1985 (2018) ✓
- 1983 (2018) ✓
- 1980 (2018) ✓
- 1981 (2018) ✓
- 1978 (2018) ✓
- 1979 (2018) ✓
- 1974 (2018) ✓
- 1977 (2018) ✓
- 1976 (2018) ✓
- 1975 (2018) ✓
- 1971 (2018) ✓

Freeze-dried chitosan matrices for slow-release of acetogenins extracted from sourop (*Annona muricata L.*) leaves

Kamarza Mulia, Farah Fauzia and Elsa Krisanti

AIP Conference Proceedings 2175, 020031 (2019);
<https://doi.org/10.1063/1.5134595>

SHOW ABSTRACT


 No Access . November 2019

A TGA-DSC of the thermal decomposition Cu-Mg catalyst precursor with various compositions

Hendriyana and Lulu Nurdini

AIP Conference Proceedings 2175, 020032 (2019);
<https://doi.org/10.1063/1.5134596>

SHOW ABSTRACT


 No Access . November 2019

The plant defence inducer activity of *Anacardium occidentale* Linn., *Azadiracta indica* A. Juss. and *Zingiber officinale* Rosc. extracts against *Cowpea mild mottle virus* infecting soybean

Wuye Ria Andayanie, Praptiningsih G. Adinurani, Wahidin Nuriana and Netty Ermawaty

AIP Conference Proceedings 2175, 020033 (2019);
<https://doi.org/10.1063/1.5134597>

SHOW ABSTRACT


 No Access . November 2019

An integrated biorefinery process of oil palm empty fruit bunch for bioethanol and flame retardant

Dian Burhani and Eka Triwahyuni

AIP Conference Proceedings 2175, 020034 (2019);
<https://doi.org/10.1063/1.5134598>

SHOW ABSTRACT


 No Access . November 2019

Prediction of the degradation route of α -cellulose hydrothermal liquefaction in super-critical organic solvents into bio-oil

Rakhman Sarwono, Andreas, Teuku Beuna Bardant and Silvester Tursiloadi

AIP Conference Proceedings 2175, 020035 (2019);
<https://doi.org/10.1063/1.5134599>

SHOW ABSTRACT

 No Access . November 2019


The reduction of hydrogen cyanide (HCN) and the measurement of antioxidant activity in bamboo shoot as the raw material for cookies

Doddy Andy Darmajana and Novianti Wulandari

AIP Conference Proceedings 2175, 020036 (2019);
<https://doi.org/10.1063/1.5134600>

SHOW ABSTRACT

- 1972 (2018) ✓
- 1973 (2018) ✓
- 1969 (2018) ✓
- 1965 (2018) ✓
- 1970 (2018) ✓
- 1968 (2018) ✓
- 1967 (2018) ✓
- 1966 (2018) ✓
- 1964 (2018) ✓
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- 1941 (2018) ✓
- 1939 (2018) ✓
- 1938 (2018) ✓
- 1937 (2018) ✓
- 1936 (2018) ✓
- 1935 (2018) ✓
- 1932 (2018) ✓

 No Access . November 2019

Moisture effective diffusivity in Kepok Kuning banana (*Musa paradisiacal formatypica*) during convective drying by considering its shrinkage phenomenon

Teuku Beuna Bardant, Arief Amier Rahman Setiawan, Rakhman Sarwono and Sri Reny Dewi Safitri

AIP Conference Proceedings 2175, 020037 (2019);
<https://doi.org/10.1063/1.5134601>

SHOW ABSTRACT


 No Access . November 2019

Performance evaluation and operation cost analysis of electrolytes application in electrocoagulation process applied to peat wastewater treatment

Ajeng Arum Sari, Nandar Suwanto, Adelia Anju Asmara, Novita Ariani, Arief A. R. Setiawan, Joko Waluyo, Muryanto and Sudarno

AIP Conference Proceedings 2175, 020038 (2019);
<https://doi.org/10.1063/1.5134602>

SHOW ABSTRACT

 No Access . November 2019

Antifungal activity of essential oil from root bark of sintok wood (*Cinnamomum sintoc Blume*) against *Pleurotus ostreatus*

Renhart Jemi, Nuwa and Elitha Octaviani

AIP Conference Proceedings 2175, 020039 (2019);
<https://doi.org/10.1063/1.5134603>

SHOW ABSTRACT


 No Access . November 2019

Formulation and characterization of betaine-based deep eutectic solvent for extraction phenolic compound from spent coffee grounds

Elsa Anisa Krisanti, Kelvin Saputra, Muhammad Maula Arif and Kamarza Mulia

AIP Conference Proceedings 2175, 020040 (2019);
<https://doi.org/10.1063/1.5134604>

SHOW ABSTRACT

 No Access . November 2019

Optimization of palmitic acid extraction from palm oil with betaine-based natural deep eutectic solvent using response surface methodology

Kamarza Mulia, Elgusta Masanari, Ida Zahrina, Bambang Susanto and Elsa Anisa Krisanti

AIP Conference Proceedings 2175, 020041 (2019);
<https://doi.org/10.1063/1.5134605>

SHOW ABSTRACT

 No Access . November 2019

In vitro release study of sambiloto (*Andrographis paniculata*) extract


- 1933 (2018) ✓
- 1931 (2018) ✓
- 1927 (2018) ✓
- 1934 (2018) ✓
- 1930 (2018) ✓
- 1928 (2018) ✓
- 1929 (2018) ✓
- 1924 (2018) ✓
- 1926 (2018) ✓
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- 1923 (2018) ✓
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- 1898 (2017) ✓
- 1907 (2017) ✓
- 1903 (2017) ✓
- 1902 (2017) ✓
- 1900 (2017) ✓
- 1899 (2017) ✓
- 1893 (2017) ✓
- 1897 (2017) ✓
- 1896 (2017) ✓

encapsulated by casein micelle as anti-diabetic herbal drug

Muhamad Sahlan, Katerina Evelyn, Diah Kartika Pratami and Kamarza Mulia

AIP Conference Proceedings 2175, 020042 (2019);
<https://doi.org/10.1063/1.5134606>

SHOW ABSTRACT


 No Access . November 2019

The use of ammonia-elphidium (A-E) index in Jakarta Bay and Semarang coastal waters, Indonesia

Ricky Rositasari, Rachma Puspitasari, Suratno and Triyoni Purbonegoro

AIP Conference Proceedings 2175, 020043 (2019);
<https://doi.org/10.1063/1.5134607>

SHOW ABSTRACT


 No Access . November 2019

Synthesis of CuO-TiO₂ nano-composite for formaldehyde degradation application

Jessica Farah, Muhammad Ibadurrohman and Slamet

AIP Conference Proceedings 2175, 020044 (2019);
<https://doi.org/10.1063/1.5134608>

SHOW ABSTRACT


 No Access . November 2019

Modified cassava flour (mocaf) wastewater treatment using electrocoagulation reactor

Muryanto Muryanto, Ishmar Balda, Eka Mardika Handayani and Ajeng Arum Sari

AIP Conference Proceedings 2175, 020045 (2019);
<https://doi.org/10.1063/1.5134609>

SHOW ABSTRACT


 No Access . November 2019

Reduction of beta-carotene with thermal activated bentonite in Illipe butter from Nanga Yen, Kalimantan Barat

Bagas Zaki Muhammad, Muhammad Arif Darmawan and Misri Gozan

AIP Conference Proceedings 2175, 020046 (2019);
<https://doi.org/10.1063/1.5134610>

SHOW ABSTRACT


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The material origin of the particulate organic matter (POM) in the Eastern Indonesian waters

A'an J. Wahyudi, Hanny Meirinawati, Hanif B. Prayitno, Suratno, Dewi Surinati and Udhi E. Hernawan

AIP Conference Proceedings 2175, 020047 (2019);
<https://doi.org/10.1063/1.5134611>

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Bioconversion of quercetin glucosides from *Dendrophthoe pentandra* leaf using *Aspergillus acueletus* LS04-3

Rizna Triana Dewi, Yasmin Ekapratwi, Andini Sundowo, Novita Ariani, Tria Yolanda and Euis Filalla

AIP Conference Proceedings 2175, 020048 (2019);
<https://doi.org/10.1063/1.5134612>

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Selective betalain impregnation from red amaranth extract onto titanium dioxide nanoparticles

Yehezkiel Steven Kurniawan, Kristine Anggraeni, Renny Indrawati and Leny Yuliaty

AIP Conference Proceedings 2175, 020049 (2019);
<https://doi.org/10.1063/1.5134613>

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Effect of maltodextrin concentration on the characteristic of phycocyanin powder as a functional food

Siti Agustina, Novi Nur Aidha and Eva Oktarina

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The effect of bleaching treatment on the mechanical strength of PP-Kenaf composite

Yuli Husnil, Ismojo, Evana Yuanita, Azmi Azis Novovic, Tina Enyta and Mochamad Chalid

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<https://doi.org/10.1063/1.5134615>

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Surface modification of poly (ethylene terephthalate) via hydrolysed erosion process

Athanasia Amanda Septevani, Gita Novi Ariani, Eryta Septa Rosa, Gusti Ayu Agung Indah Cahyani and Muhammad Arifuddin Fitriady

AIP Conference Proceedings 2175, 020052 (2019);
<https://doi.org/10.1063/1.5134616>

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Physicochemical properties of pudding powder as a complementary food fortified with the essential mineral

Dita Kristanti and Ainia Herminiati

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Method development for determination of trace organochlorine pesticides residues in a water matrix by using gas chromatography mass spectrometry (GCMS) method

Dillani Putri Ramadhaniyngtas and Nurhani Aryana

AIP Conference Proceedings 2175, 020054 (2019);
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Synthesis and characterization of modified marine algal biomass using zero-valent iron (ZVI) particle for Cd and Pb removal

Fitri Budiyanto, Ajeng Arum Sari, Lestari, Sandi Permadi, Asep Bayu, Harmesa and Deny Yogaswara

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Bioremediation of oil-contaminated sediment by hydrocarbonoclastic bacterial consortium immobilized in different types of carrier

Kathleen Irena Junusmin, Brian Saputra Manurung and Yeti Darmayati

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<https://doi.org/10.1063/1.5134620>

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The effect of carbonization temperature and activator to the characteristics of melinjo shell (*Gnetum genom*) activated carbon

Apriliana Dwijayanti, Safril Kartika and Yuliusman

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The effect of chitosan into paper properties in papermaking

Muryeti, Faraqh Eka Pratiwi, Risqi Tri Yuniastuti and Estuti Budimulyani

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Effect of time alkali treatment on chemical composition and tensile strength properties of kenaf single fibers

Ismojo, E. Yuanita, E. M. Rosa, L. Calvin and M. Chalid

AIP Conference Proceedings 2175, 020059 (2019);
<https://doi.org/10.1063/1.5134623>

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Crystalinity index evaluation of *Dendrocalumus asper* fibers through variation of chemical treatment

E. Yuanita, Ismojo, H. K. Adi and M. Chalid

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<https://doi.org/10.1063/1.5134624>

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Comparison study on morphology and mechanical properties of starch, lignin, cellulose - based polyurethane foam

E. Kustiyah, D. A. Setiaji, I. A. Nursan, W. N. Syahidah and M. Chalid

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Selective optical chemosensors of Fe³⁺ ions using 1*H*-indole-2,3-dione

Muhammad Riza Ghulam Fahmi, Yehezkiel Steven Kurniawan, Leny Yuliaty and Hendrik O. Lintang

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Improvement of Spaghetti composite quality based on local flours (mocaf, corn, rice and soybean)

Nur Kartika Indah Mayasti, Mirwan Ushada and Makhmudun Ainuri

AIP Conference Proceedings 2175, 020063 (2019);
<https://doi.org/10.1063/1.5134627>

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Physicochemical and pasting properties of composite flours for making gluten-free bread

Lista Eka Yulianti, Nok Afifah and Riyanti Ekafitri

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Certified reference materials for calibration of conductivity meter at the measuring of electrolytic conductivity in water: Preparation and its measurement

Nuryatini Hamim, Fransiska Sri Herwahyu Krismastuti, Ayu Hindayani and Yosi Aristiawan

AIP Conference Proceedings 2175, 020065 (2019);
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Concentration, spatial distribution, and source apportionment of polycyclic aromatic hydrocarbons (PAHs) in marine surface sediments from Cirebon coastal water, West Java, Indonesia

Khozannah, Deny Yogaswara, Ita Wulandari, Edward, Dwi Hindarti and Dede Falahudin

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Charge analysis of monomer hydrogenated graphene

Gagus Ketut Sunnardianto

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Synthesis hybrid bio-polyurethane foam from biomass material

Achmad Nandang Roziafanto, Made Subekti Dwijaya, Rima Yunita, Majid Amrullah and Mochamad Chalid

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<https://doi.org/10.1063/1.5134632>

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Supplementation of ginger and cinnamon extract into goat milk kefir

Fitri Setiyoningrum, Gunawan Priadi and Fifi Afiati

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Sol-gel method for synthesis of Li⁺-stabilized Na-β"-alumina for solid electrolytes in sodium-based batteries

Anisa I. Agustina, Karl Skadell, Cornelius L. Dirksen, Matthias Schulz and Samuel P. Kusumocahyo

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Effect of reaction temperature and sodium tripolyphosphate (STPP)/ starch ratio on phosphorylation of sweet potato (*Ipomoea batatas* L.) starch

Asaf Kleopas Sugih, Amelia Dewi, Devina Yukano and Henky Mujana

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Effect of hydrogen gas pressure on biofuel characteristics in hydrogenation reaction of non-oxygenated fraction of bio-oil

Dijan Supramono, Elizabeth Verdiana Listiono and Mohammad Nasikin

AIP Conference Proceedings 2175, 020072 (2019);
<https://doi.org/10.1063/1.5134636>

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Combination of ozone, H₂O₂, and adsorption using granular activated carbon for shampoo synthetic wastewater treatment

Fidelis Ayodya Amba, Natasha Vidi Salsabila and Eva Fathul Karamah

AIP Conference Proceedings 2175, 020073 (2019);
<https://doi.org/10.1063/1.5134637>

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Synthetic shampoo liquid waste treatment with ozonation: Case study with peroxone and activated carbon

Natasha Vidi Salsabila, Fidelis Ayodya Amba, Eva Fathul Karamah and Setijo Bismo

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Cellulose triacetate synthesis from empty fruit bunches of oil palm's cellulose

Aniek Sri Handayani, Chrisvynlia, Theo Doohan, Marcelinus Christwardana and Enjarlis

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Effect of stirring speed on characteristics of biofuel in catalytic hydrogenation of non-oxygenated bio-oil

Dijan Supramono, Billi and Mohammad Nasikin

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Modification of synthetic carpet using chitosan-titania nanocomposite for self-cleaning purposes

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Mohamad Iman Sulaeman, Muhammad Ibadurrohman and Slamet

AIP Conference Proceedings 2175, 020077 (2019);
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Formulation and evaluation of serum from red, brown and green algae extract for anti-aging base material

Melati Septiyanti, Lilis Liana, Sutriningsih, Bayu Kumayanjati and Yenny Meliana

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In vitro selection of effective microbial strains for bioaugmentation on oil polluted sediment from Cilacap coastal, Indonesia

Yeti Darmayati and Lies Indah Sutiknowati

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The effect of calcination temperature on the synthesis of magnetic silica nanoparticles from geothermal sludge

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