

Preparation of Green-Emissive Zinc Oxide Composites Using Natural Betacyanin Pigment Isolated from Red Dragon Fruit

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Abstract: In this work, we reported the synthesis of green-emissive composite materials of zinc oxide (ZnO) and isolated betacyanin pigment from red dragon fruit (RDF) extract utilizing organic linkers, i.e. (3-chloropropyl)trimethoxysilane (CPTMS) and (3-aminopropyl)trimethoxysilane (APTMS). Betacyanin was extracted using a maceration technique, while CPTMS-ZnO and APTMS-ZnO were prepared by mixing ZnO and the respective organic linker in ethanol. The obtained ZnO/CPTMS and APTMS-ZnO composites were separately added into the RDF extract, followed by stirring at room temperature for 24 h. As high as 80 and 90% of betacyanin was successfully impregnated onto CPTMS-ZnO and APTMS-ZnO, respectively. A comparison study was made by preparing RDF-CPTMS and RDF-APTMS first and then introducing them onto ZnO. In this case, as high as 81 and 100% of betacyanin in RDF-CPTMS and RDF-APTMS, respectively, were impregnated onto ZnO. These results revealed that APTMS was a better organic linker than CPTMS and the order of the steps to introduce APTMS was important. The presence of betacyanin on the composite materials was confirmed by FTIR and fluorescence spectroscopy. All the composite materials had an excitation signal at 426–428 nm and emission signals at 459 and 517–518 nm, demonstrating their promising application as green-emissive materials.

Keywords: betacyanin; composite; green-emissive; red dragon fruit; ZnO

■ INTRODUCTION

In recent years, there has been a high demand for research related to photonic materials due to its wide application for human appliances [1-5]. Regarding this topic, many researches are still focused on enhancing and optimizing the efficiency of the materials. Zinc oxide (ZnO), a material with a direct bandgap of 3.3 eV, has been used over the past several years as photocatalysts, semiconductors, electronic material, etc. [6-7]. With such a large bandgap, pure ZnO can only be applied in the UV region [8]. Therefore, enhancing the efficiency of ZnO material in the visible region leaves a challenge for material scientists. Sensitizing ZnO material with either synthetic dye or natural pigment is a well-known procedure to enhance the light absorption region of this

material [9-10]. However, synthetic dye generates serious environmental problems in their application to produce dye-sensitized materials [11].

Red dragon fruit (RDF) is an abundantly available tropical fruit in Indonesia, which contains several unique secondary metabolites, such as betacyanin, flavonoid, and carotenoid [12-14]. It has been reported that betacyanin pigment could be isolated from the flesh of RDF using a maceration method for 24 h [15]. Incorporation of natural pigments may overcome the limitation of the ZnO material as a photocatalyst to be utilized in the visible region, especially in the red color region as the main part of the visible region in the solar spectrum. Betacyanin is recognized as one of the major red natural pigments, and therefore, very potential to be

used to widen the light absorption of ZnO. Owing to the alcohol and carboxylic acid functional groups, it is possible to immobilize the betacyanin pigment onto the surface of ZnO materials using a suitable linker agent. Even though the visible light emissive material is promising, the method to prepare the ZnO composites consisting of the betacyanin pigment from RDF has not been developed yet. We demonstrated that the use of a suitable organic linker would be a great method to prepare the composite. This research opens the possibility to utilize natural pigments to improve the properties of the photonic materials such as ZnO.

In the present work, we also reported that the prepared betacyanin-modified ZnO exhibited strong green emission, which would be very interesting for photonic material application. We prepared green emissive ZnO based composite materials utilizing isolated betacyanin pigment from RDF and either (3-chloropropyl)trimethoxysilane (CPTMS) or (3-aminopropyl)trimethoxysilane (APTMS) as the organic linker agent. By a stirring method for 24 h, four (4) types of ZnO based materials, named as RDF/CPTMS-ZnO, RDF/APTMS-ZnO, RDF-CPTMS/ZnO and RDF-APTMS/ZnO have been successfully obtained based on the use of different linkers and the order to introduce the betacyanin pigment on the ZnO. These materials were thoroughly characterized by spectroscopy instruments to investigate the physicochemical properties as green emissive composite materials.

■ EXPERIMENTAL SECTION

Materials

Fresh RDFs were obtained commercially from a supermarket in Malang, East Java. Meanwhile, the ZnO materials were prepared in a similar manner to the procedure which has been reported previously [16]. The organic linkers (3-chloropropyl)trimethoxysilane (CPTMS) and (3-aminopropyl)trimethoxysilane (APTMS) were purchased from Sigma Aldrich. Ethanol was purchased from E-Merck in pro analysis grade and used without any further purifications.

The optical properties of the composite materials were measured by diffuse reflectance ultraviolet-visible

(DR UV-Vis) spectrophotometer (JASCO V-760), while the bandgap energy of the composite materials was calculated from Tauc plot using the data. The infrared and fluorescence spectra of the composite materials were recorded by Fourier transform infrared spectrophotometer (JASCO FTIR-6800) and spectrofluorometer (JASCO FP-8500), respectively.

Procedure

Isolation of betacyanin pigment from the RDF

The isolation of betacyanin pigment from RDF was carried out according to the previously described method [15,17]. In short, the flesh of RDF (5.0 g) was dried at room temperature overnight and macerated with distilled water (50 mL) for 24 h. Afterward, the mixture was filtered. The clear filtrate was finally characterized by FTIR spectrophotometer and spectrofluorometer.

Preparation of composite materials

In this work, there were two types of composite materials, namely RDF/linker-ZnO and RDF-linker/ZnO materials. The term RDF/linker-ZnO material showed that the organic linker was reacted first with ZnO and then further reacted with RDF extract, while the term RDF-linker/ZnO material indicated that the organic linker was first reacted with RDF extract and then further reacted with ZnO.

The RDF/linker-ZnO materials, i.e., RDF/CPTMS-ZnO and RDF/APTMS-ZnO, were prepared through a simple stirring method. First, the organic linker (0.25 mL) and ZnO (0.25 g) were added stepwise into ethanol (12.5 mL), and then the mixture was stirred at 500 rpm at room temperature for 24 h. After that, the residue was filtered and added to 5% v/v RDF extract in ethanol (12.5 mL). The mixture was stirred for another 24 h. The mixture was then filtered and the residue was dried to obtain the RDF/linker-ZnO composite materials.

The RDF-linker/ZnO materials, i.e. RDF-CPTMS/ZnO and RDF-APTMS/ZnO were also simply obtained by a stirring method. First, the organic linker (0.25 mL) was added dropwise to 5% v/v RDF extract in ethanol (12.5 mL) and the mixture was stirred at 500 rpm for 24 h. Afterward, ZnO (0.25 g) was added

and the mixture was stirred for 24 h. The mixture was filtered and the residue was dried to obtain the RDF-linker/ZnO composite materials.

■ RESULTS AND DISCUSSION

Preparation of Composite Materials

In this work, ZnO-based composite materials were prepared and evaluated for their potential application as emissive materials. At first, the betacyanin pigment was isolated from RDF extract through a maceration method with distilled water. As has been reported, betacyanin pigment was confirmed to be successfully isolated from RDF extract using the maceration method [15,18-19]. On the other hand, the prepared ZnO has been reported to give a bandgap energy value of 3.29 eV [16].

The isolated betacyanin pigment was used to prepare the composites in two different procedures. In the first procedure, the organic linker was attached first on the surface of the ZnO material and then the isolated betacyanin pigment was immobilized onto it. Meanwhile, the other way involved the reaction between the isolated betacyanin pigment and organic linker, followed by its immobilization onto the surface of the ZnO material.

The color appearances of the prepared ZnO based materials can be seen in their photographs as shown in Fig. 1. From Fig. 1(a), it can clearly be observed that ZnO has a white color. In contrast, both RDF/CPTMS-ZnO and RDF-CPTMS/ZnO were obtained as bright purplish

solids as shown in Fig. 1(b) and (c), respectively. On the other hand, Fig. 1(d) and (e) showed that RDF/APTMS-ZnO appeared as a pale yellow solid while RDF-APTMS/ZnO appeared as a bright yellow solid. The difference of the color appearance showed the different forms of immobilized betacyanin on the composite material. It was previously reported by Strack et al. (2003) that the color of betacyanin would depend on the pH of the solution [20]. Below pH 10, the color of betacyanin solution is red, while its color turns to yellow when the pH is higher than 10. Since the CPTMS organic linker is acidic, the pH of the filtrate during the reaction was 4.53. Therefore, the color of RDF/CPTMS-ZnO and RDF-CPTMS/ZnO was purplish. On the other hand, the use of APTMS caused the mixture to be alkaline (pH = 10.8), hence the color of betacyanin pigment changed from red to bright yellow. Therefore, it is reasonable for both RDF/APTMS-ZnO and RDF-APTMS/ZnO to be obtained as yellow solids. The formation of such color in the composites showed that the light absorption of ZnO had been expanded successfully to the visible light region.

Characterization of Composite Materials

DR UV-vis spectra of ZnO and the composite materials are shown in Fig. 2. As expected, the white ZnO sample only showed absorption in the UV region up to 380 nm. On the other hand, the composite materials gave extra absorption at the background level in

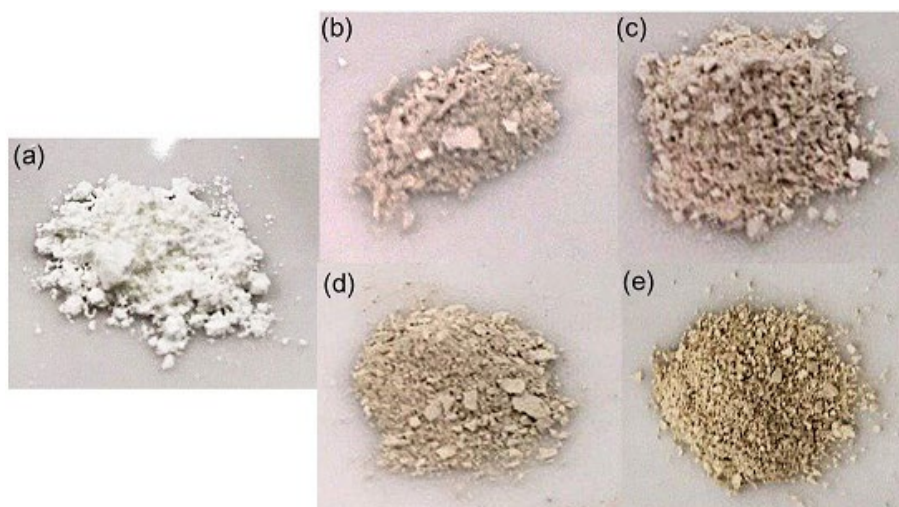


Fig 1. Photographs of (a) ZnO and ZnO based composite materials, including (b) RDF/CPTMS-ZnO, (c) RDF-CPTMS/ZnO, (d) RDF/APTMS-ZnO, and (e) RDF-APTMS/ZnO

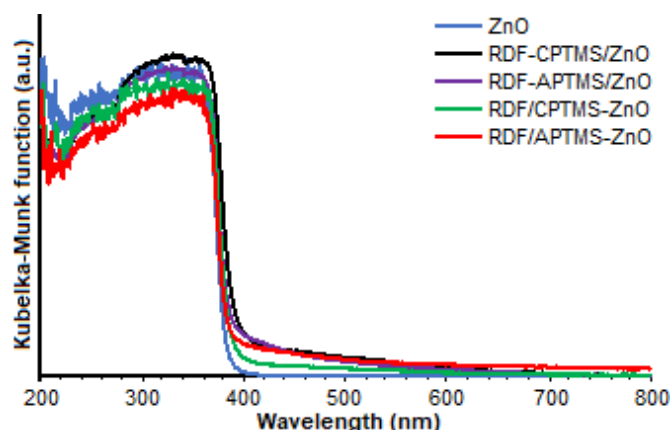


Fig 2. DR UV-vis spectra of ZnO and ZnO based composite materials

the visible region due to the presence of betacyanin from the RDF extract, which is in good agreement to the color appearances of the composites. From the DR UV-vis spectra of the composite materials, the bandgap energy of the composite materials was calculated accordingly and shown in Table 1. The bandgap energy of unmodified ZnO, RDF-CPTMS/ZnO, RDF-APTMS/ZnO, RDF/CPTMS-ZnO and RDF/APTMS-ZnO composite materials were 3.29, 3.22, 3.23, 3.26 and 3.26 eV, respectively. The bandgap energy of the composite materials was not significantly different from the unmodified ZnO because the betacyanin pigment was only immobilized on the surface of the ZnO materials. In this case, the betacyanin pigment would act as the sensitizer. The values of the bandgap energy in this work were close to other reported ZnO dye-sensitized solar cells

[21] and natural pigment-modified ZnO materials prepared from laali [22].

The bandgap energy of the composite materials seemed to be independent on the organic linker, but dependent on the order of betacyanin introduction. For example, when the organic linker was immobilized first onto the ZnO surface, the bandgap energy was 3.26 eV for both CPTMS and APTMS. However, when the organic linker-betacyanin was immobilized onto the ZnO surface, the bandgap energy was slightly lower, which were 3.22 and 3.23 eV for CPTMS and APTMS, respectively. When betacyanin was reacted first with the organic linker, the betacyanin-linker might have created new energy states that were more positive than the conduction band of ZnO and the linker or betacyanin itself, which in turn gave a slightly lower bandgap energy than ZnO and RDF/linker-ZnO.

The FTIR spectra of ZnO, RDF extract, and composite materials are shown in Fig. 3. The FTIR spectrum of unmodified ZnO showed two main absorption peaks at 3394 and 434 cm^{-1} , corresponding to Zn-O-H and Zn-O-Zn moieties, respectively [23].

Table 1. Bandgap energy of the composite materials

Materials	Bandgap (eV)
ZnO	3.29
RDF-CPTMS/ZnO	3.22
RDF-APTMS/ZnO	3.23
RDF/CPTMS-ZnO	3.26
RDF/APTMS-ZnO	3.26

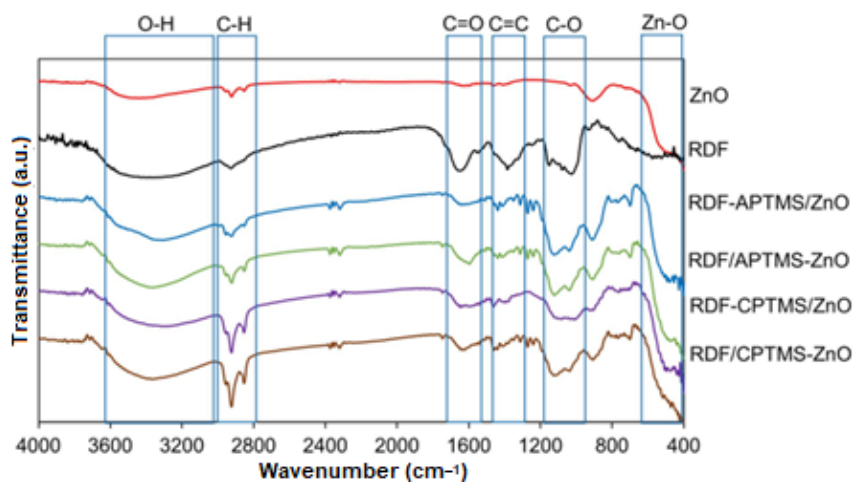


Fig 3. FTIR spectra of ZnO, RDF, and ZnO based composite materials

The FTIR spectrum of isolated betacyanin from the RDF extract showed O-H, C-H sp^3 , C=O, C=C, and C-O functional groups at 3340, 2902–2891, 1628, 1512–1314, and 1024 cm^{-1} , respectively [19]. As depicted in the FTIR spectra of the composite materials, in addition to the ZnO absorption peaks, the peaks of betacyanin functional group also existed, strongly indicating that the

immobilization of betacyanin pigment on the surface of the ZnO material has been successfully carried out.

Further characterization of composite materials was investigated by fluorescence spectroscopy. The three-dimensional fluorescence spectra of unmodified ZnO, RDF extract, and the composite materials are shown in Fig. 4. The unmodified ZnO gave a strong excitation

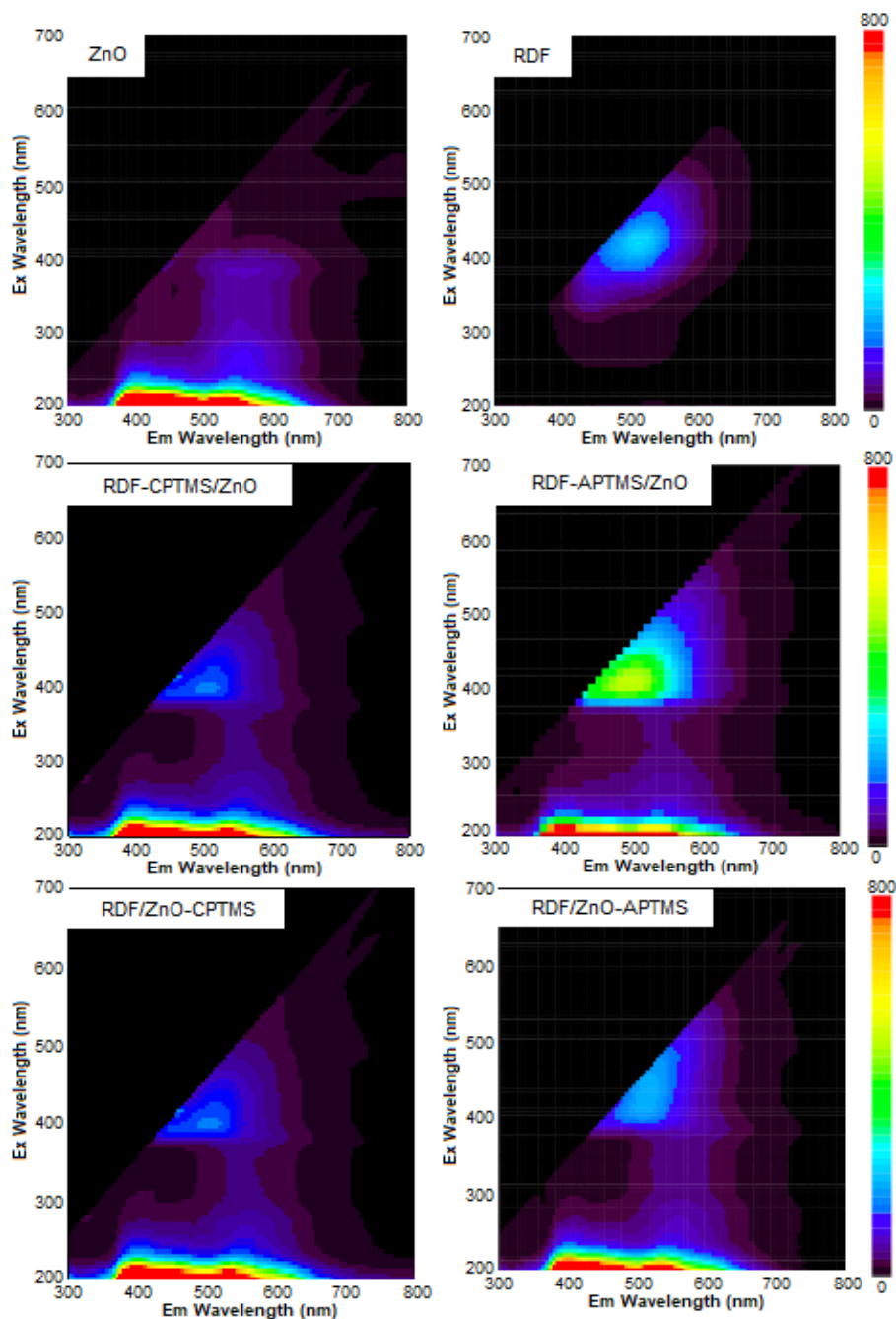


Fig 4. Three-dimensional fluorescence spectra of ZnO, RDF, and ZnO based composite materials

peak around 200 nm in good agreement with the strong light absorption of ZnO in the UV region and the weak excitation peak could be observed around 428 nm. The emission region of ZnO was wide and in the range of 350–650 nm. On the other hand, the RDF extract gave a weak excitation peak around 405 nm and the emission region was in the range of 430–680 nm. It was obvious that the three-dimensional fluorescence spectrum of composite materials was indeed a combination of the

three-dimensional fluorescence spectrum of ZnO and RDF extract. This result again clearly indicated the successful immobilization of the RDF extract onto ZnO.

From the fluorescence spectrum in the visible region of ZnO (Fig. 5), it was confirmed that the wavelengths of the excitation and emission were at 428 and 459 nm, respectively. These wavelengths were different from those of the RDF extract, where the maximum wavelengths of the excitation and emission

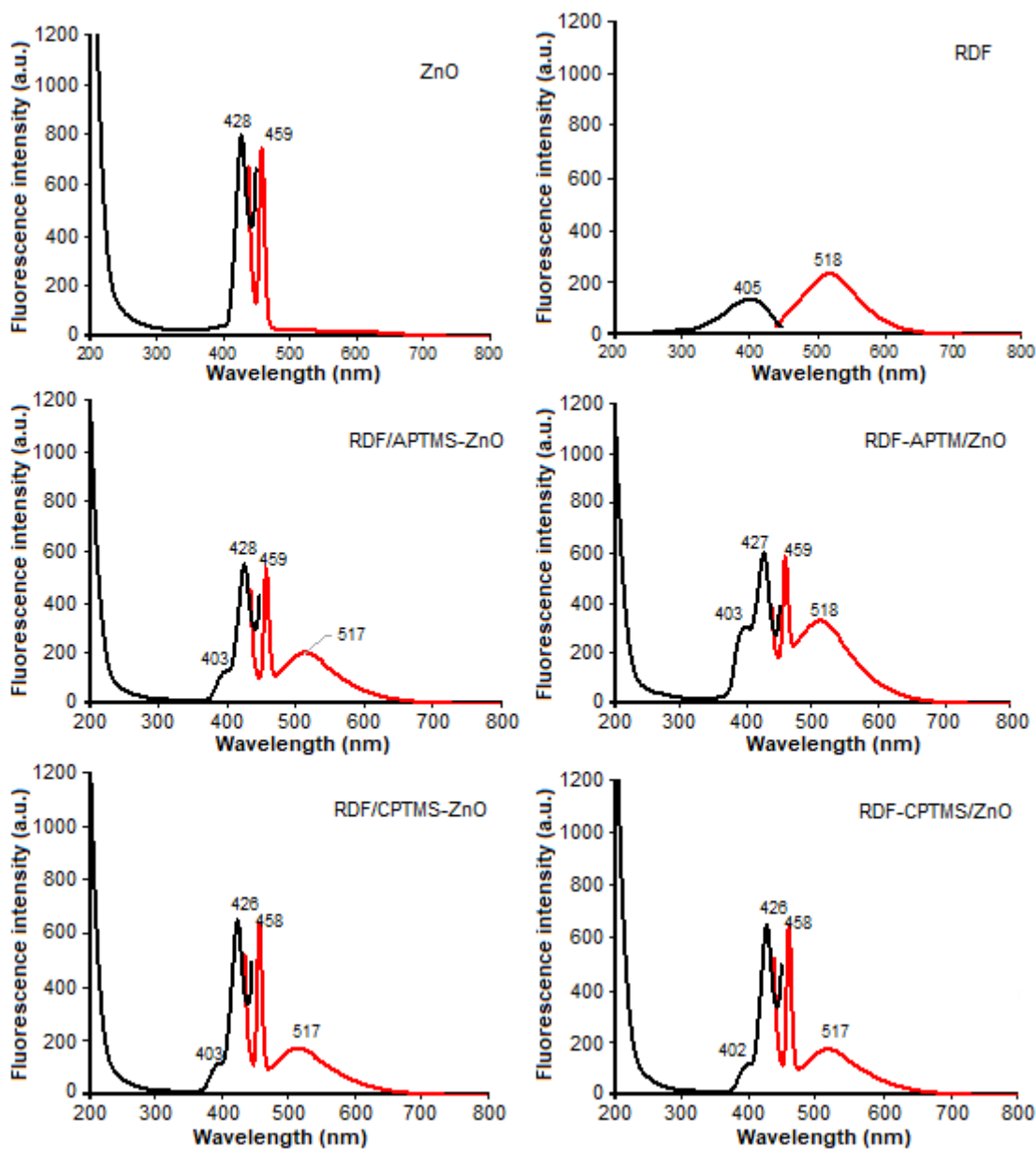


Fig 5. Fluorescence spectra of ZnO, RDF, and ZnO based composite materials. Black and red lines represent excitation and emission spectrum, respectively

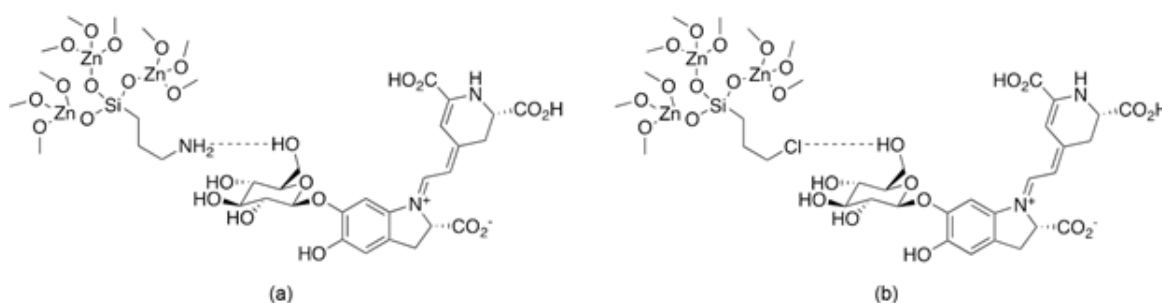


Fig 6. Proposed interactions between betacyanin pigment with (a) APTMS and (b) CPTMS on the ZnO surface

spectrum were found at 405 and 518 nm, respectively. As shown in Fig. 4, all the excitation and emission peaks of ZnO and RDF extract also existed on the composite materials. This work demonstrated that the immobilization of betacyanin pigment onto the ZnO surface has successfully enhanced the absorption region of the composite materials. Since the emission signals were found in 458–518 nm region, the prepared composite materials are promising to be used as green-emissive materials.

From the fluorescence intensity of the emission peak at 518 nm, the immobilization percentage of betacyanin pigment on the composite materials was calculated and shown in Table 2. The immobilized betacyanin amounts on the RDF-CPTMS/ZnO and the RDF-APTMS/ZnO were found to be 81% and 100%, respectively. This result showed that APTMS was a better organic linker than CPTMS in obtaining the ZnO composite. As betacyanin pigment has alcohol and carboxylic acid groups, betacyanin pigment would interact well with the amino substituent of APTMS through the hydrogen bondings rather than with the chloro substituent of CPTMS. This would be the reason why the immobilization percentages of betacyanin pigment were higher on the APTMS-based composite than the CPTMS-based one. The proposed interactions between the betacyanin pigment and the organic linker on the ZnO surface are shown in Fig. 6. Similar to our previous work [24], the interactions would mainly happen between either amino or chloro substituent of the organic linker and the primary alcohol substituent on the tetrahydropyran ring of the betacyanin due to less steric hindrance. Therefore, it was reasonable that flavonoid and carotenoid pigments were not immobilized

Table 2. Immobilized betacyanin percentages on the ZnO based composite materials

Materials	Immobilized betacyanin (%)
ZnO	0
RDF-CPTMS/ZnO	81
RDF-APTMS/ZnO	100
RDF/CPTMS-ZnO	80
RDF/APTMS-ZnO	90

on the ZnO materials because these pigments are non-polar [25], and thus, could not interact through hydrogen bondings with the linker agents.

On the other hand, only 80% and 90% of betacyanin was impregnated onto CPTMS-ZnO and APTMS-ZnO, respectively. While this result also showed that APTMS was better than CPTMS to impregnate the betacyanin onto ZnO, the order to introduce APTMS was found to be critical to obtaining larger amounts of immobilized betacyanin. When the betacyanin pigment was reacted with APTMS-ZnO material, it is possible for the betacyanin pigment to interact weakly with the Zn-OH group. Because of that, after filtration the immobilization percentage of betacyanin pigment on the RDF/APTMS-ZnO was lower than that of the RDF-APTMS/ZnO material.

■ CONCLUSION

In the present work, we successfully prepared four types of ZnO based composite materials through immobilization of betacyanin pigment on the surface of ZnO material using either CPTMS or APTMS as the organic linker. Both the RDF/CPTMS-ZnO and RDF-CPTMS/ZnO were obtained as purplish solids, while the RDF/APTMS-ZnO and RDF-APTMS/ZnO materials

appeared as pale yellow and bright yellow solid, respectively. The bandgap energy of the composite materials was not significantly different from the unmodified ZnO because the betacyanin pigment was immobilized on the surface of the ZnO. The FTIR spectra of the composite materials showed the presence of O–H, C–H sp^3 , C=O, C=C, and C–O functional groups. As high as 100% immobilized betacyanin could be achieved when the betacyanin was reacted first with APTMS, followed by impregnation onto ZnO. This result showed the importance of using APTMS and the order to introduce the betacyanin pigment onto ZnO. From the fluorescence spectrum of the composite materials, the emission signals were found at 458–518 nm region, demonstrating that the prepared composite materials are promising to be used as green-emissive materials.

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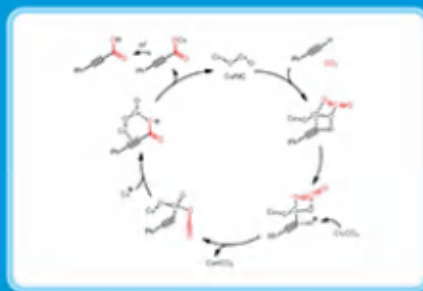
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Vol 21, No 1 (2021)

Accredited by RISTEK-BRIN No.: 85/M/KPT/2020 (April 1, 2020)



Table of Contents

Articles

Hydrothermal Synthesis: Low-Temperature Subcritical Water for Ceria-Zirconia Mixed Oxides Preparation

1-13

Siti Machmudah, Widiyastuti Widiyastuti, Wahyudiono Wahyudiono, Sugeng Winardi, Hideki Kanda, Motonobu Goto

10.22146/ijc.47357 Abstract views : 472 | views : 488

Antifungal Activity of TiO₂/Ag Nanoparticles under Visible Light Irradiation

14-23

Nahzim Rahmat, Endang Tri Wahyuni, Adhitasari Suratman

10.22146/ijc.49150 Abstract views : 527 | views : 273

Performance Improvement of Tetraethylorthosilicate Consolidated Andesite Rock by Adding Titanium Tetraisopropoxyde and Silica Particles

24-36

Nahar Cahyandaru, Endang Tri Wahyuni, Nuryono Nuryono

10.22146/ijc.49786 Abstract views : 322 | views : 251

Acid-Alkaline Treatment of Mordenite and Its Catalytic Activity in the Hydrotreatment of Bio-Oil

37-45

Febi Yusniyanti, Wega Trisunaryanti, Triyono Triyono

10.22146/ijc.51496 Abstract views : 507 | views : 459

Polynomial Regression Analysis for Removal of Heavy Metal Mixtures in Coagulation/Flocculation of Electroplating Wastewater

46-56

Siti Wahidah Puasa, Kamariah Noor Ismail, Muhammad Amarul Aliff Bin Mahadi, Nur Ain Zainuddin, Mohd Nazmi Mohd Mukelas

10.22146/ijc.52251 Abstract views : 365 | views : 313

Preparation of Green-Emissive Zinc Oxide Composites Using Natural Betacyanin Pigment Isolated from Red Dragon Fruit

57-65

Yehezkiel Steven Kurniawan, Hendrik Oktendy Lintang, Leny Yuliaty

10.22146/ijc.52351 Abstract views : 712 | views : 370

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INFORMATION


- [▶ For Readers](#)
- [▶ For Authors](#)
- [▶ For Librarians](#)




CoMFA, Molecular Docking and Molecular Dynamics Studies on Cycloguanil Analogues as Potent Antimalarial Agents 66-76 

 Isman Kurniawan, Muhammad Salman Fareza, Ponco Iswanto


 10.22146/ijc.52388  Abstract views : 477 |  views : 536




Synthesis and Characterization of Copper Impregnated Mesoporous Carbon as Heterogeneous Catalyst for Phenylacetylene Carboxylation with Carbon Dioxide 77-87 


 Putri Nurul Amalia, Iman Abdullah, Dyah Utami Cahyaning Rahayu, Yuni Krisyuningsih Krisnandi

 10.22146/ijc.52778  Abstract views : 488 |  views : 373




Synthesis and Characterization of Sodium Silicate Produced from Corncobs as a Heterogeneous Catalyst in Biodiesel Production 88-96 

 Alwi Gery Agustan Siregar, Renita Manurung, Taslim Taslim

 10.22146/ijc.53057  Abstract views : 1403 |  views : 539




Degradation of Methylene Blue Using Cadmium Sulfide Photoanode in Photofuel Cell System with Variation of Electrolytes 97-107 

 Gunawan Gunawan, Abdul Haris, Didik Setiyo Widodo, Linda Suyati, Wilman Septina

 10.22146/ijc.53131  Abstract views : 638 |  views : 281




Bismuth Oxide Prepared by Sol-Gel Method: Variation of Physicochemical Characteristics and Photocatalytic Activity Due to Difference in Calcination Temperature 108-117 


 Yayuk Astuti, Brigita Maria Listyani, Linda Suyati, Adi Darmawan

 10.22146/ijc.53144  Abstract views : 638 |  views : 384




Assessing the Effect of Weak and Strong Acids as Electrolytes in the Removal of Cesium by Soil Electrokinetic Remediation 118-127 



 Rudy Syah Putra, Alfi Ihda Amalia, Naila Zahrotul Jannah

 10.22146/ijc.53283  Abstract views : 320 |  views : 276

FTIR-based Fingerprinting and Chemometrics for Rapid Investigation of Antioxidant Activity from *Syzygium polyanthum* Extracts 128-136 


 Eti Rohaeti, Fadila Karunina, Mohamad Rafi

 10.22146/ijc.54577  Abstract views : 439 |  views : 433




Ligand Based Pharmacophore Modeling, Virtual Screening, and Molecular Docking Studies of Asymmetrical Hexahydro-2H-Indazole Analogs of Curcumin (AIACs) to Discover Novel Estrogen Receptors Alpha (ER α) Inhibitor 137-147  

 Hariyanti Hariyanti, Kusmadi Kurmardi, Arry Yanuar, Hayun Hayun


 10.22146/ijc.54745  Abstract views : 466 |  views : 440 |  views : 180

Analysis of Dissolution of Salicylamide from Carrageenan Based Hard-Shell Capsules: A Study of the Drug-Matrix Interaction 148-156 


 Muhammad Al Rizqi Dharma Fauzi, Esti Hendradi, Pratiwi Pudjiastuti, Riyanto Teguh Widodo

 10.22146/ijc.55047  Abstract views : 428 |  views : 586




Conformational Analysis of Diterpene Lactone Andrographolide towards Reestablishment of Its Absolute Configuration via Theoretical and Experimental ECD and VCD Methods 148-156  

 Muhamad Faid A Kadir, Agustono Wibowo, Fatimah Salim, El Hassane Anouar, Khalijah Awang, Moses Kiprotich Langat, Rohaya Ahmad

 10.22146/ijc.55206  Abstract views : 333 |  views : 294 |  views : 178

Synthesis, Characterization, and Study of Proton Exchange Polymer Membrane Properties of Sulfonated Copolymer Eugenol-diallyl Phthalate 168-178 

 Ngadiwiyana Ngadiwiyana, Nor Basid Adiwibawa Prasetya, Gunawan Gunawan, Tutuk Djoko Kusworo, Heru Susanto


 10.22146/ijc.55353  Abstract views : 447 |  views : 334

Nicotiana tabacum Mediated Green Synthesis of Silver Nanoparticles and Ag-Ni Nanohybrid: Optical and Antimicrobial Efficiency 179-191  

 Anuoluwa Abimbola Akinsiku, Joseph Adeyemi Adekoya, Enock Olugbenga Dare

 10.22146/ijc.56072  Abstract views : 620 |  views : 377 |  views : 173

Determination of Eugenol in Personal-Care Products by Dispersive Liquid-Liquid Microextraction Followed by Spectrophotometry Using *p*-Amino-*N,N*-dimethylaniline as a Derivatizing Agent 192-200  

 Bahaa Malik Altahir, Omar Abdulazeez, Sarmad Bahjat Dikran, Keith Edward Taylor

 10.22146/ijc.56198  Abstract views : 470 |  views : 203 |  views : 184

KEYWORDS

HPLC QSAR TiO₂

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ATOM 1.0

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<p>Design of New Quinazoline Derivative as EGFR (Epidermal Growth Factor Receptor) Inhibitor through Molecular Docking and Dynamics Simulation</p> <p> Herlina Rasyid, Bambang Purwono, Harno Dwi Pranowo</p> <p> 10.22146/ijc.57012 Abstract views : 325 views : 285</p>	201-211
<p>Separation of Rhodamine B Dye from Aqueous Media Using Natural Pomegranate Peels</p> <p> Zainab Mohammad Saigl, Arwa Mohammed Ahmed</p> <p> 10.22146/ijc.58592 Abstract views : 385 views : 252</p>	212-224

Short Communication

<p>The Use of Real-Time Polymerase Chain Reaction Combined with Specific-Species Primer for Analysis of Dog Meat DNA in Meatball</p> <p> Abdul Rohman, Wiranti Sri Rahayu, Sudjadi Sudjadi, Sudiby Martono</p> <p> 10.22146/ijc.48930 Abstract views : 230 views : 321</p>	225-233
<p>Statistical Study on the Interaction Factors of Polypropylene-Graft-Maleic Anhydride (PP-g-MA) with Graphene Nanoplatelet (GNP) at Various Poly(Lactic Acid)/Polypropylene (PLA/PP) Blends Ratio</p> <p> Farah Hafidzah, Mohd Bijarimi, Waleed Alhadadi, Suriyati Salleh, Mohammad Norazmi, Erna Normaya</p> <p> 10.22146/ijc.54036 Abstract views : 319 views : 354</p>	234-242
<p>Computer-Aided Discovery of Pentapeptide AEYTR as a Potent Acetylcholinesterase Inhibitor</p> <p> Enade Perdana Istyastono, Vivitri Dewi Prasasty</p> <p> 10.22146/ijc.55447 Abstract views : 216 views : 252</p>	243-250
<p>Study of Mangosteen Peel (<i>Garcinia mangostana</i> L.) Waste Capability to Recover Au(III) and Ag(I) in Aqueous Solution</p> <p> Mellia Harumi, Rian Kurniawan, Agustiwandina Saputri, Dian Hanna Saraswati, Meissha Ayu Ardini, Sri Sudiono</p> <p> 10.22146/ijc.57670 Abstract views : 360 views : 319</p>	251-258