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SINTESIS DAN KARAKTERISASI $FeTiO_3$ DARI LIMBAH BATERAI DENGAN METODE SOL-GEL UNTUK PENANGANAN METILEN BIRU SECARA FOTOKATALISIS

Rizky Dinda Islami[1], Eko Prabowo Hadisantoso[2], Vina Amalia[3]
Department of Chemistry, Universitas Islam Negeri Sunan Gunung Djati, Bandung, Indonesia
*Corresponding Author email: rdindaislami@gmail.com

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Limbah baterai Zn-C termasuk bahan berbahaya dan beracun (B3). Hal ini dapat memberikan dampak buruk pada ekosistem lingkungan dan makhluk hidup. Maka dari itu limbah ini perlu ditangai dengan baik agar keberadaannya tidak membahayakan lingkungan. Salah satu caranya dengan memanfaatkan kembali logam besi dari jaket luar baterai dan mereaksikannya bersama bahan semikonduktor TiO_2 membentuk logam oksida jenis perovskit yaitu $FeTiO_3$. Penelitian ini bertujuan untuk mensintesis senyawa $FeTiO_3$ yang merupakan material semikonduktor dengan potensi kinerja fotokatalisis untuk menangani limbah zat warna metilen biru karena tingkat kestabilan yang tinggi dalam media air di bawah iradiasi sinar tampak. Senyawa $FeTiO_3$ disintesis dengan metode sol-gel dengan media pengkhelat larutan asam sitrat dan dikalsinasi selama 2 jam pada suhu 600ºC. Hasil karakterisasi dengan X-ray Diffraction (XRD) menunjukkan bahwa struktur $FeTiO_3$ adalah rhombohedral dengan fasa ilmenit, ukuran kristal sebesar 133 nm dan nilai kristalinitas sebesar 63%, namun terdapat pengotor Fe_3O_4 magentit. Pada hasil Scanning Electron Microscope (SEM) menunjukkan morfologi $FeTiO_3$ berbentuk bulat tidak beraturan yang tersebar secara acak dengan permukaan berpori dan algomerasi. Sedangkan, energi celah pita $FeTiO_3$ beradsarkan hasil analisis UV/Vis-DRS yang diperoleh dengan metode Kubelka Munk yaitu 1,97 eV. Berdasarkan kinerja kondisi terbaik fotokatalisis dalam degradasi metilen biru didapat persen degradasi sebesar 97% dengan massa optimum 90 mg selama 180 menit pada konsentrasi 10 ppm dan pH 11.

30, and 45 seconds, which aimed to identify the characteristics and analyze the performance of the capsule shell. The results of the organoleptic characterization of the capsule shell are odorless, transparent brownish, rigid, and elastic. The capsule shell specifications meet the standards, except for the sample weight exceeding ±0.08 grams from the standard capsule. The degree of swelling of the capsule shell with variations in dipping time of 15, 30, and 45 seconds is 14.20%, 10.04%, and 7.46%. The functional group analysis of the capsule shell confirms the materials used. This functional group provides a wave number shift that still covers the bond wave number range and indicates the presence of *compound interactions in the material formulation in the capsule shell sample. Disintegration time with variations in dipping time of 15, 30, and 45 seconds are 3 hours, 4 hours, and 5 hours. The disintegration time obtained exceeds the disintegration time limit set by Pharmacopoeia VI edition, namely less than 15 or 30 minutes. The dissolution test, at the 360th minute, resulted in dissolution percentages of 92.72%, 79.56%, and 63.55%. These dissolution percentages do not meet the standard set by Pharmacopoeia VI, which requires a dissolution rate of ≥ 85% within 30 minutes. Based on the research results, the entire formula of starch-carrageenan with a combination of $CaCl_2$ and glycerol can be formed into a capsule shell. Where the capsule shell evaluation results are almost close to pharmacopoeial standard VI, namely the sustained release type*

INTRODUCTION

Biopolymers, characterized by macromolecular biomolecules with intricately linked monomer units, stand out from synthetic polymers due to their well-defined three-dimensional structure. The term "bio" emphasizes their biological origin and inherent degradability. Mimicking synthetic polymer properties, biopolymers, particularly polysaccharides, exhibit promise in gas and vapor sensors, boasting biocompatibility and unique attributes. Moreover, their sustainability, renewability, and minimal environmental impact make them ideal for various applications, notably industrial wastewater treatment.

The categorization of biopolymers into polysaccharides, polynucleotides, and polypeptides highlights their diverse nature. Polysaccharides, abundant in nature, include well-known examples like starch, cellulose, glycogen, chitin/chitosan, pectin, and alginate. Within polysaccharides, subcategories such as sugar-based, starch-based, cellulose-based, and lignin biopolymers further enhance their versatility.

Polysaccharides, derived from plants, animals, and microorganisms, possess unique properties like biodegradability and non-toxicity, making them valuable in food packaging. Starch, cellulose, chitin, chitosan, alginate, and hyaluronic acid are commonly employed in food packaging applications, forming biopolymer-based nanoparticles that enhance physical properties.

Exploring nanocomposites, polysaccharide-based nanocomposites (PNCs) have emerged as biodegradable packaging materials, leveraging nanoparticles like titanium oxide, zinc oxide, silver, gold, and copper oxide for added antibacterial properties. Citric acid plays a crucial role in crosslinking with polysaccharides, enhancing properties like thermal stability and water resistance. The esterification reaction between citric acid and starch, under both acidic and alkaline conditions, contributes to the formation of a three-dimensional network, impacting the polymer's strength and molecular weight.

Adding citric acid to biodegradable starches improves various properties, such as thermal stability, water resistance, and flowability, while impacting elastic strain and starch recrystallization. The concentration of citric acid during cross-linking is influenced by factors like reaction temperature and pH. Despite numerous studies on bioplastics based on polysaccharide biocomposites using cross-linked citric acid, there's a limited bibliometric perspective. Bibliometric analysis becomes crucial for understanding scientific trends and advancements, helping researchers navigate and contribute to the ever-evolving landscape of biopolymer research.

EXPERIMENT

1.1 Data Based Search

In the quest for information on "Thermal Characterization of Biopolymers-Based Polysaccharide Bionanocomposite With Modified Cross-Linking Using Citric Acid," Google Scholar served as the primary search tool. The preference for Google Scholar was driven by its extensive coverage of article publications and the accessibility of journals without cost. To streamline the search and identify relevant articles, the Publish or Perish tool was employed. The search spanned from 2019 to 2023, and keywords such as "Bioplastic," "Crosslinked," "citric acid," and "thermal characterization" were utilized. Subsequently, the search outcomes were compiled and saved in *csv format for further analysis.

1.2 Data Extraction and Screening

Once the articles were retrieved, they underwent categorization based on various parameters, including author names, citation counts, article titles, publication years, media type (journal, proceedings), seminar or book section details, journal names, publishers, and abstracts. Following this categorization, the data was formatted into RIS (Research Information Systems) for subsequent analysis in Vosviewer. Simultaneously, the information was also extracted in CSV (Comma-Separated Values) format for further analysis using Microsoft Excel. This multi-format extraction allowed for comprehensive examination and exploration of the collected data through diverse analytical tools.

1.3 Visualization Data

Following the acquisition of data in CSV format, the next step involves conducting an analysis using Microsoft Excel. This analysis aims to generate graphs illustrating the trends in the publication of related articles, the fluctuation in citation counts, and the identification of prolific authors contributing the most articles each year. Simultaneously, the files saved in RIS format are subjected to analysis in Vosviewer to explore network visualization, overlay visualization, and density visualization. These complementary analyses provide a comprehensive understanding of the publication landscape, author contributions, and citation dynamics within the specified research domain.

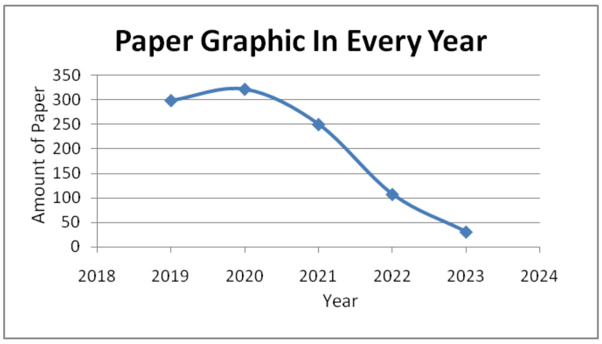
1.4 Data Analysis

Upon collecting data from both Microsoft Excel and Vosviewer, a thorough analysis ensued. This examination focused on tracking the yearly progression of article outcomes, exploring the connections between authors and publishers, identifying the most prolific authors, and visualizing the derived clusters. By delving into these aspects, the analysis aimed to unravel the intricate dynamics of the research landscape, shedding light on the evolution of articles over time, author-publisher relationships, prominent contributors, and the thematic clusters visualized through the analysis.

RESULT AND DISCUSSION

Publication Trend

The findings are centered on data from 1000 relevant articles published between 2019 and 2023, focusing on bioplastics, crosslinked, citric acid, and thermal characterization. Figure 2 illustrates a diminishing trend in research growth over the past five years. The most significant surge in publications occurred in 2020, boasting 322 publications, constituting a 32.2% increase. In contrast, 2023 stands out as the year with the least growth, witnessing only 30 publications, equivalent to a 3% increase. The trajectory of publications on bioplastics, specifically those based on polysaccharide bionanocomposites with crosslinked citric acid, exhibited an upswing in 2020 by 32.2%, followed by a gradual decline leading up to 2023.



Graphic 1. documentspublished “Bioplastics”, bionanocomposites”, acid” and “thermal ch

1.2 Top Article Document by The Most Citation

The top 10 documents with the most citations are in the table. The highest number of citations was 745 with the title "Evolution and Synthesis of carbon dots: from carbon dots to carbonized polymer dots" by C. Xia, et., al., 2019.

N o	Cites	Author	Title	Year	Journal Sour	Publisher So

					ces	urces
	745	C. Xia, et., al.	Evolution and Synthesis of carbon dots: from carbon dots to carbonized polymer dots	2019	Advanced Science	Wiley Online Library
	481	M C Catoira, et., al	Overview of Natural hydrogels for regenerative medicine aplication	2019	Journal of Materials	Springer
	316	N A N egm .,	Advancement of modification of chitosan	2020	International Jo	Elsevier

		et., al	biopolymer and its potential application		urnal	
	302	K. Im., et., al	An Introduction to performing Immunofluorescemce Staining	2019	Biobanking	Springer
	295	R. Curvello., et., al	Engineering Nanocellulose hydrogels for biomedical application	2019	Advanced in Colloid	Elsevier
	270	M. Guerre., et., al	Vitrimers: Directing Chemical Reactivity to Control Material	2020	Chemical Sciences	Pubs.rsc.org

			Properties			
7	227	S.H. Zainal., et., al	Preparation of Cellulose-based Hydrogels: Review	2021	Journal of Material	Elsevier
8	219	Q. Li., et., al	Tannic Acid-Polyethylenimine Crosslinked Loose Nanofiltration Membrane for Dye/Salt Mixture Separation	2019	Journal of Membrane	Elsevier
9	218	L.A. Heinrich	Future Opportunities Bio-Based Adhesives-Advantages	2019	Green Chemistry	Pubs.rsc.org
			Beyond Renewability			
10	214	X. Yang. Et., al	A Fully Bio-Based Epoxy Vitrimer: Self-Healing, Triple-Shape Memory and Reprocessing Triggered by Dynamic Covalent Bond Exchange	2020	Material & Design	Elsevier

Tabel 1. Top Article Document by The Most Citation and Publisher

The quantity of citations within a journal serves as an indicator of the article's quality. A higher citation count typically signifies a superior quality article. Citations play a crucial role in reinforcing findings within a scientific work, expressing appreciation for prior authors, informing readers about relevant research, elucidating concepts, discussing applied theories, and providing a comprehensive list of references (Tee, K. S., 2017). The representation of the number of publishers within an article is depicted in the image.

Graphic 2. Total Publisher

1.3 Co-authorship Analysis of Author

The following author visualization shows that the authors are most active in the following research topics shown in the figure 1.

Figure 1. Co-Authorship Analysis

The image reveals the presence of five distinct clusters distinguished by different colors. Cluster 1, highlighted in red, is dominated by the most active author, Zhang, Y.

Cluster 2, represented in green, features Li, Y as the most prolific author. Cluster 3, in a beer-colored shade, showcases Wang, Y as the most active contributor. Meanwhile, Cluster 4, marked in yellow, is led by the most active author, Chen, J. Lastly, Cluster 5, depicted in purple, is spearheaded by the most active author, Li, J. Analyzing these clusters, it becomes evident that the most active authors in this domain are Zhang Y, Wang Y, and Li Y.

1.4 Visualization of Research Topic Area using VOSviewer

OSviewer serves as a powerful software tool designed for generating graphs from network data and facilitating their exploration and analysis. Its functionalities can be outlined as follows:

1. Map Creation: VOSviewer allows the creation of maps based on existing networks or the option to generate a network from scratch. This can encompass various elements such as scientific publications, journals, researchers, and state research institutes. Additionally, it enables the formation of networks based on keywords or terms, establishing connections through bibliographic links or citation links to co-authors. Data from sources like Web of Science, Scopus, PubMed, RIS, or Crossref JSON files can be employed to construct these networks.

2. Map Exploration: VOSviewer provides three distinct graphic views—network view, overlay view, and density view. The zoom and scroll functions are instrumental in fully exploring the map, especially when dealing with extensive maps containing a multitude of objects.

While VOSviewer is primarily designed for bibliometric network analysis, its versatility extends to creating and exploring graphs based on a wide range of network data (Nees Jan Vack, 2023).

1.4.1 Network Visualization

In VOSviewer, network visualization emerges as a robust approach to delve into the relationships among various elements in a dataset, encompassing co-authorship networks, co-citation networks, or keyword co-occurrence networks. This visualization tool proves invaluable for unraveling intricate relationships within a research dataset, aiding in the identification of key contributors, emerging trends, and collaborative domains. It's important to note that interpreting these results may necessitate domain knowledge and a nuanced understanding of the specific research context.

Network visualization is represented by lines or networks connecting one term to another. As depicted in Figure 2, there are five clusters distinguished by different colors, totaling 62 items. Within these clusters, there are 15 nodes with the main node in cluster 1 being "Membrane," 15 nodes with the main node in cluster 2 being "Carboxylic Acid," 14 nodes with the main node in cluster 3 being "Biopolymer," 12 nodes with the main node in the cluster "Boric Acid," and in cluster 5, there are 7 nodes with the main node having a low frequency of occurrence of the keyword "Adsorbent." The infrequent occurrence of this keyword suggests potential areas for future research and exploration.

Figure 2. Network Visualization

1.4.2 Overlay Visualization

Overlay visualization in VOSviewer enhances network visualization by incorporating additional layers of information, thereby enriching the analysis with various attributes or metrics. In Figure 3, a relationship is depicted between cluster analysis and keyword terms, aiming to discern the trending topics from those that are less explored, alongside the corresponding publication years. Notably, as of 2020, terms such as "Crosslinking Reaction," "Biopolymer," "Pectin," and "Polyvinyl Alcohol" have not been extensively researched, indicating potential areas for further exploration.

Figure 3. Overlay Visualization

1.4.3 Density Visualization

Density visualization in VOSviewer facilitates the examination of element concentration within a network visualization, aiding in the identification of clusters or regions with varying density levels for elements like authors or keywords. By observing the color hue of each cluster, you can gauge the density of clusters in your network. Figure 4 demonstrates that the keywords "Crosslinking Reaction," "Acetic Acid," and "PVA" exhibit higher density. This suggests that there is room for further exploration and development in research on crosslinked polymers, indicating that certain aspects of this domain may not have been thoroughly investigated.

Figure 4. Density Visuallization

CONCLUSION

This section contains the final conclusion in an outline using interconnected paragraphs rather than per point.

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Briefly describe the people/institutions involved and supporting the research. For example: financing or contributing the results of the analysis, and others.

REFERENCES

The reference must be written in IEEE style. Within the text, The author's name is listed as first initial, last name. Example: Adel Al Muhairy would be cited as A. Al Muhairy (NOT Al Muhairy, Adel).

Note that in the case of three or more authors, only the last name of the first author is cited and the others are denoted by et al. In the References chapter, you should write based on the order of appearances, not alphabetically. Example of References:

[1] P. Bormans, *Ceramics are more than Clay Alone*. Cambridge: Cambridge International Science Publishing, 2004.

[2] M. P. Fewell, "The atomic nuclide with the highest mean binding energy," *American Journal of Physics*, vol. 63, no. 7, pp. 653-658, 1995.

The minimum number of references is 20, with 80% of references cited from academic journals within not more than the last 10 years.