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Synthesis and characterization of chitosan/polyvinyl alcohol crosslinked poly(*N*-isopropylacrylamide) smart hydrogels via γ-radiation

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ABSTRACT

Chitosan-based smart hydrogels with stable mechanics and good flexibility have many desirable qualities and broad applications. In this study, novel smart hydrogels of chitosan crosslinked with poly(*N*-Isopropylacrylamide) (pNIPAAm) and polyvinyl alcohol (PVA) were synthesized through freezing and thawing procedures followed by gamma (γ)-radiation at room temperature. The effect of γ-irradiation dose on the gel fraction and water absorption characteristics of chitosan-pNIPAAm hydrogels was investigated. In addition, the structure–property behaviour of the hydrogels was characterized using Fourier Transform Infra-Red (FTIR) spectroscopy and thermogravimetric analysis (TGA). The experimental results revealed that the hydrogels synthesized with a 15-kGy total dose showed a higher gel content (83.73%) compared to 5-kGy and 10-kGy total doses, while the appropriate dose of γ-radiation to achieve the highest absorption in water (484.146%) was 5-kGy. The hydrogel characterization test confirmed that cross-linking occurred between pNIPAAm, PVA, and chitosan. A higher the radiation dose resulted in more cross-links in the hydrogel and resulted in better thermal stability. This study confirmed that chitosan/PVA cross-linked pNIPAAm hydrogel with dual pH/temperature stimuli-responsiveness holds promise for various applications.

1. Introduction

Hydrogels are three-dimensional cross-linked polymeric networks having the capability to hold a large amount of aqueous solvents and biological fluids within their structures [1]. Hydrogels have emerged as smart materials and have shown great potential in many applications because of their features which are responsive to various external stimuli [2]. Hydrogels as stimuli-responsive polymers display a sharp transition in physicochemical characteristics under a small change in environmental conditions, such as pH, temperature, ionic strength, light exposure, magnetic field, electrical field, ultrasound, etc [3]. With an array of triggering mechanisms, these stimuli-responsive hydrogels allow precise control over basic material properties, such as physical structure, porosity, swelling, and modulus [4]. Their ability to change their conformation and properties when stimulated at different conditions causes them to be used prominently in wastewater treatment [5], sustained release of agrochemicals [6], food science [7], and biomedical field [8].

Smart hydrogels prepared from polysaccharides have many advantages because of their attractive characteristics including biocompatibility, biodegradability [9], self-healing, and responsiveness to environmental stimuli [10]. Among them, chitosan is an excellent excipient because it is an abundant and natural polysaccharide which has many amazing properties such as low toxicity, high biocompatibility, desirable biodegradability [11], and antimicrobial activity [12]. Chitosan which is a derivative of chitin, a natural polymer extracted from crab and shrimp shells, exhibits pH sensitivity and polycationic properties. The external and environmental pH affects the swelling and shrinking behaviour of chitosan-based hydrogel derived from the protonation/deprotonation of the primary amine group (NH_2) [13]. Because of its responsive properties, chitosan is to become an advanced biopolymer in the development of smart polymeric delivery systems [3].

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Chitosan has hydrophilic (—OH) and hydrophobic (—NH_2) groups which can be easily modified with other natural or synthetic polymers through physically or chemically cross-linked reactions [14] to produce hydrogels with different stimulus responses. In addition to the pH, the temperature is also one of the most exploited stimuli in 3-dimensional networks because it is easy to measure, set and use in testing [13], so the dual responsive chitosan-based hydrogel (pH and temperature) has become a tremendous development in recent years [15,16,17]. To improve its performance, chitosan can be modified by crosslinking with other moieties/monomers to increase its sensitivity to pH and temperature [18]. The group of thermoresponsive polymers includes poly(*N*-isopropylacrylamide) [16,17,19], poly(N,*N*-diethylacrylamide) [20], and poly (*N*-vinylcaprolactam) [21].

Poly(*N*-isopropylacrylamide)(pNIPAAm) is a thermoresponsive polymer which is applied in a wide range of promising applications because of its well-defined structure and characteristics especially its temperature sensitivity which is close to a lower critical solution temperature (LCST) [22]. Thus, the pH sensitivity of chitosan and the volume phase transition temperature of pNIPAAm have attracted the interest of various scientists to develop materials as carriers and controller the release of active substances [16,23].

A relatively simple and inexpensive technique, that does not involve the use of toxic chemical agents and allows simultaneous sterilization of crosslinked polymers, is radiation induction [24]. A previous study used radiation as the crosslinking agent to produce graft copolymers [25], and another previous study used the irradiation method to reduce and control polysaccharides' molecular weight distribution to adjust their water solubility and gelling ability [26]. To increase the swelling ability and mechanical strength of the hydrogel, modification of the radiation method in combination with other methods such as freeze-thawing has been successfully carried out by several researchers. γ-irradiation combined with freeze-thawing was applied to synthesize pH-responsive hydrogels from poly(vinyl alcohol) (PVA) and water-soluble chitosan (ws-chitosan) [27], while PVA/carboxymethyl-chitosan (CM-chitosan)/honey hydrogels were prepared using radiation technique and a combinational method (radiation followed by freeze-thawing) [28].

In this work, novel dual responsive hydrogels from chitosan and *N*-isopropylacrylamide (pNIPAAm) with the addition of polyvinyl alcohol (PVA) were fabricated by introducing the combination method of freeze-thawing and γ-irradiation to induce crosslinking. Reports on swelling studies of hydrogels having a dual response to pH and temperature from a combination of 3 ingredients namely chitosan, pNIPA and PVA prepared using the freeze-thawing method followed by gamma radiation have not been found. The influence of γ-radiation dose on the gel content, the swelling properties, and the hydrogel thermal characteristics was examined in this study.

2. Materials and methods

2.1. Material

Chitosan (industrial grade, deacetylation degree of 85 %, Biotech Surindo), poly(*N*-isopropylacrylamide) (pNIPAAm; Aldrich), polyvinyl alcohol (PVA; Merck), acetic acid (glacial, Merck), pH buffer (3, 5, 9), and distilled water were used throughout the experiment.

2.2. Preparation of pH- and thermo-responsive hydrogels

A total of 0.5 g of chitosan was added into 50 ml of 1 % (v/v) acetic acid solution under a stirring condition to form a chitosan solution. Meanwhile, distilled water as much as 100 ml was added into a bottle containing 5 g of PVA, then the bottle was closed and heated in an autoclave at 120 °C for 30 min. PVA solution was then brought out from the autoclave and cooled at room temperature. After that, 10 ml of PVA solution was mixed with 10 ml of chitosan solution. Then, 0.5 g of pNIPAAm (0.5 g) was added to the mixed solution and then followed by a stirring process at room temperature. The final solution as much as 5 ml was taken and kept in 5-ml tubes and continued with 3 cycles of freezing and thawing procedure, where each cycle was run for 24 h (12 h for freezing and 12 h for thawing). After the freezing and thawing procedure, the samples were γ-irradiated under a constant rate of irradiation dose (2.5 kGy/min) at room temperature. The total dose of γ-irradiation for each sample was varied to 5, 10, and 15 kGy. After the irradiation process, the physical samples turned into gels. At this stage, the resulting gels are known as hydrogels. Then, the hydrogels were cut into small sizes (approximately 20 mm^2) and characterized.

2.3. Testing and characterization

2.3.1. Transform Infra-Red (FTIR)

The samples were characterised using FTIR (Shimadzu IR – Prestige 21) to confirm the presence of functional groups of chitosan, pNIPAAm, and PVA in the hydrogels.

2.3.2. Thermo gravimetric analysis (TGA)

In this study, the TGA was examined to find the thermal properties of synthesized hydrogels. During the heating process, there was decreasing in the hydrogel weight due to thermal degradation. The thermal characteristics of the hydrogels were measured using DTG-60 TA WS-60 Shimadzu at a temperature in the range of 20 – 600 °C.

2.3.3. Swelling study

The swelling behaviour of the non-crosslinked and crosslinked gels was examined by swelling the gels in media at different pHs and temperatures. Pre-weighed dry hydrogels were immersed in pH-5 buffer solutions at a temperature of 30 °C for 24 h. The gels were withdrawn from the solutions at different time intervals and their wet weights were determined after the withdrawn gels were absorbed for the first time with filter papers and immediately weighed. The swelling ratio was determined using the equation (1) based on the study reported by Erizal [29].

$$Swelling\ \ ratio\ \ (\%) = \frac{W_s}{W_d} \times 100\% \tag{1}$$

Where swelling ratio also can be named as the water absorption (% wt) of the gels, Wd and Ws are the sample weights in the dry and swollen states, respectively.

2.3.4. Gel content measurement

Hydrogel powder as much as 0.01 g (W_0) was packed in the tea bag. Then, the tea bag containing the hydrogel powder was immersed in distilled water for 24 h at room temperature. After that, the tea bag was dried in an oven for 24 h at 60 °C and weighed (W_1). Gel content was determined using the equation (2).

$$Gel\ \ content\ \ (\%) = \frac{w_1}{w_0} \times 100\% \tag{2}$$

where w_1 is the dried gel weight (g) and w_o is the initial gel weight (g).

2.3.5. SEM study

The superabsorbent hydrogel samples were soaked for 24 h until a swelling equilibrium was reached and then frozen for 24 h. The samples were dried using a freeze dryer at −105 °C and then coated with a thin layer of gold and analyzed by using a Scanning Electron Microscope (SEM) EDX Carl Zeiss EVO MA10.

3. Results and discussion

3.1. Structure analysis

Chemical modifications induced by the irradiation process are analyzed using FTIR. The spectra of nonirradiated and irradiated hydrogels are showed in Fig. 1 and marked with arrows. The FTIR shows peak shifts caused by interactions between two or more polymer mixtures used through hydrogen bonding, formation of coordinate bonds, or any other complex. The spectra that emerge from an FTIR image can produce the same characteristic features as individual polymers or sometimes several of the band shifts from their original position [30].

In the spectrum of PVA, the peaks are observed at 3402.4 cm^{-1}, corresponding to the O—H groups. The absorbance at 2960.73 cm^{-1} can be attributed to the CH stretching, and the absorbance at 1004.96 cm^{-1} corresponds to the C═O stretching. In the spectrum of pNIPAAm, the absorption bands at 3280.92 cm^{-1}, 2920.35 cm^{-1}, 1546.91 cm^{-1}, and 1170.24 cm^{-1} are attributed to stretching vibration absorption peaks of –NH, C—H, C═O, and C—N, respectively. The spectrum of chitosan shows the peak at 3442.94 cm^{-1} which is attributed to O—H and N—H, the peak at 2920.23 cm^{-1} which corresponds to C—H, and the absorbances at 1589.34 cm^{-1}, 1255.45 cm^{-1}, and 1154.25 cm^{-1} which are attributed to C═O, C—O, and C—N, respectively. When the mixture of chitosan-pNIPAAm-PVA is irradiated, the grafted hydrogel is produced, and its FTIR spectrum is presented in a curved shape. The irradiated hydrogel spectrum confirms the N—H & O—H groups at a wavelength of 3471.87 cm^{-1}, C—H groups at a wavelength of 2924.09 cm^{-1}, C═O groups at a wavelength 1633.71 cm^{-1}, the C—N group at a wavelength of 1267.23 cm^{-1} and the C—O group at a wavelength of 1045.42 cm^{-1}.

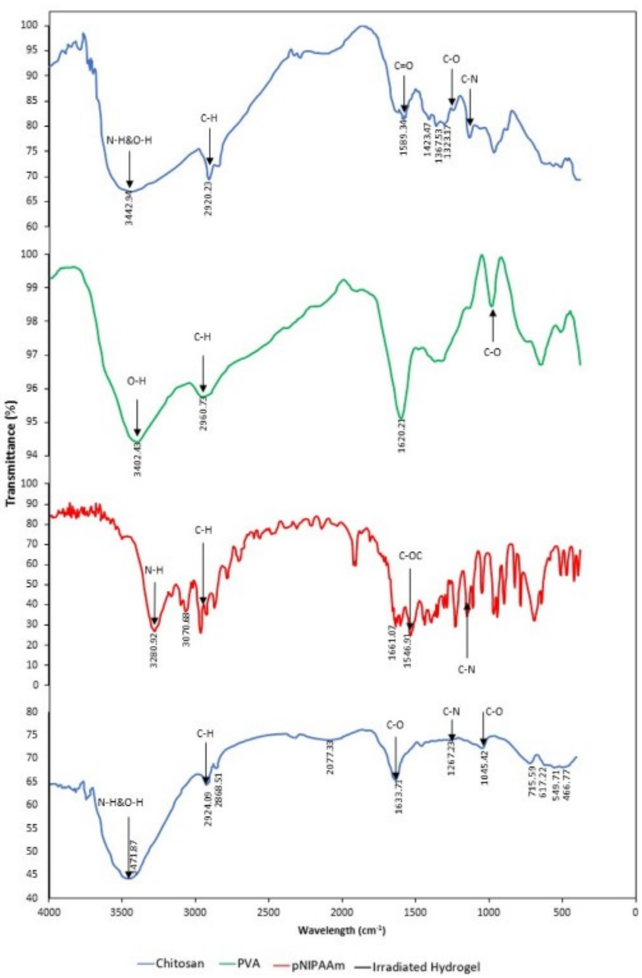

Fig. 1. FTIR spectra.

It is found that the spectrum of the hydrogel has the same peaks as the peaks of the spectra of chitosan, pNIPAAm, and PVA, but the intensity of the peak at 3471.87 cm^{-1} is stronger compared to that of the same peak in the spectra of chitosan and pNIPAAm. The O—H and N—H groups in the hydrogel have a long, tapered and broad spectrum shape with relatively strong intensity, different from the O—H groups in PVA and chitosan, and the N—H groups in pNIPAAm and chitosan which tend to be a short curve shape with medium intensity. It confirms that chitosan, pNIPAAm, and PVA are crosslinked to form an interpenetrating network (IPN) which causes the accumulation of O—H and N—H groups so that the spectral form is longer with stronger intensity [17,31,32].

A material which comprises two or more networks which are fully or partially interlaced on a molecular scale but not covalently bonded to each other and cannot be separated unless chemical bonds are broken is called an IPN. If one polymer is linear and penetrates another cross-linked network without any chemical bonds between them, the IPN is called a semi-IPN. If both of the polymers are crosslinked, it is called a full-IPN [31]. Crosslinking reaction mechanism for hydrogel formation using γ-irradiation in solution has been extensively reported [27,33,34]. The radical polymerization reactions that occur in solution include initiation, propagation, and termination steps. In the initiation stage, the water molecules decompose to form hydrogen radicals (H +) and hydroxyl radicals (OH–). Hydroxyl radicals are predicted to have the most important role in the hydrogel formation reaction because they are able to break the bonds in the polymer to form polymer free radicals [27]. In addition, the formation of polymer radicals can also directly occur due to γ-irradiation [35]. The radicals that are formed are then cross-linked to each other at the propagation stage and finally produce a stable product at the termination stage.

The combination of chitosan, pNIPAAm, and PVA as materials for synthesizing hydrogels suggests the possibility of cross-linking with IPN. This is based on the results of previous studies, where a hydrogel production showed a physical cross-linking between chitosan and PVA. The bond that occurs between chitosan and PVA was in the form of hydrogen bonds [36]. On the other hand, the combination of pNIPAAm and PVA produced a semi-IPN hydrogel in which pNIPAAm was cross-linked in the presence of the hydrophilic PVA [31]. Semi IPN and full IPN hydrogels were also successfully prepared by combining chitosan with pNIPAAm [37]. The FTIR spectra, as can be observed in Fig. 1, confirms the similarity spectra pattern of all the material used compared to the hydrogel formed, with a stronger intensity in the hydrogel product. Thus, the interaction between chitosan, pNIPAAm, and PVA structures in the hydrogel network are mainly in the form of hydrogen bonds.

The thermograms (Fig. 2) show changes in the weight of a compound as a function of temperature and time. The Fig. 2 (A) shows the thermogram of PVA. The PVA as much as 10 mg is heated at the temperature range of 20 °C to 500 °C for 2 h. In the beginning, the PVA sample shows small weight loss due to water evaporation at ± 100–200 °C, and then the sample weight is continuously degraded. Significant weight loss of PVA is performed at a temperature of 260 °C until 500 °C. The melting point of PVA is 200 °C. Based on TGA (DTG-60) analysis, the biggest value of PVA weight loss is –3.02 mg min^{-1} which is obtained at a temperature of 370.13 °C. Furthermore, the Fig. 2(B) shows the thermogram of

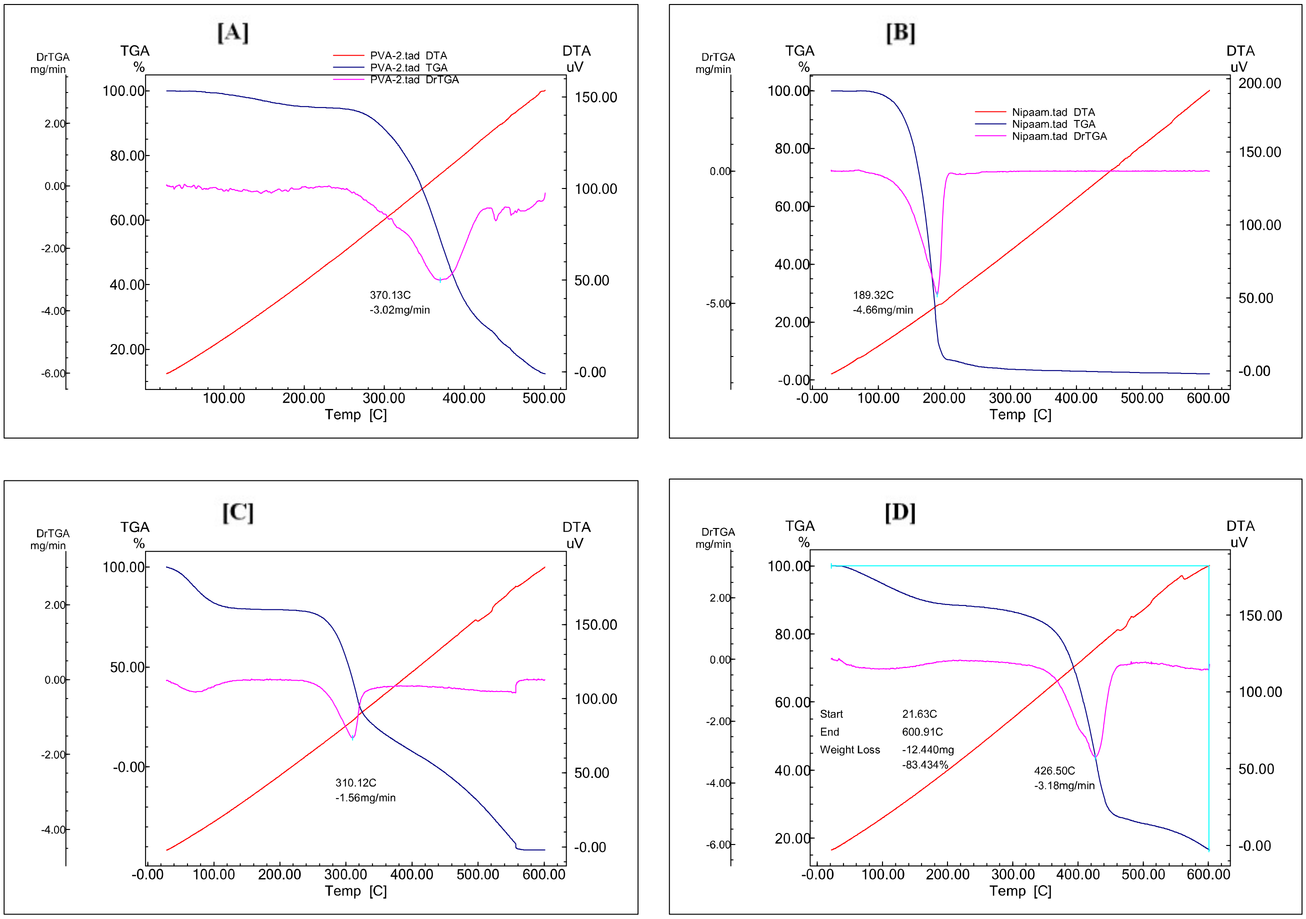

Fig. 2. Thermograms of the reactants and the irradiated hydrogel: A) PVA, B) pNIPAAm, C) chitosan, D) irradiated hydrogel.

pNIPAAm. The pNIPAAm as much as 10 mg is heated from 25 °C to 600 °C for 115 min. At the heating temperature range of ± 100–2 00 °C, the weight of pNIPAAm decreases. Based on TGA (DTG-60) analysis, the biggest pNIPAAm weight degradation occurs at a temperature of 189.32 °C with a degradation rate of -4.66 mg min^{-1}. The Fig. 2(C) shows the thermogram of chitosan.The chitosan as much as 10 mg is heated from 25 °C to 600 °C for 115 min. Water evaporation occurs in the chitosan sample at a heating temperature of ± 30–100 °C. The significant decrease in chitosan weight is performed at 260 °C and the weight starts to be constant at 560 °C. Based on TGA (DTG-60) analysis, the biggest value of chitosan weight loss is -1.56 mg min^{-1} which is obtained at 310.12 °C. Moreover, after the thermal process, the weight of hydrogel resulting from γ-irradiation dose of 5 kGy decreases by -83.434 %, as shown in Fig. 2 (D). That value represents the number of hydrogels that are degraded as a result of heat treatment.

The thermal decomposition characteristics of pNIPAAm and chitosan shown in Fig. 2 (B) and Fig. 2 (C) are similar to the results published in a previous study on pH and thermosensitive hydrogels, where the weight of pNIPAAm was reduced at higher temperature treatment [38]. Another previous study also proved that the introduction of PVA made the thermal decomposition of chitosan hydrogel change to a higher temperature [39]. The weight loss of the hydrogel due to heat treatment is smaller than the weight loss of its raw materials such as chitosan, pNIPAAm, and PVA. The high value of the hydrogel weight loss is correlated to the impurities contained in the samples and the crosslinking homogeneity of the polymer. These results reveal that the radiation causes a rapid cross-linking and affects the increase in the polymer's thermal stability indicating difficult decomposition and a high flame-retardant effect [40].

3.2. Effect of irradiation dose on the gel contents and swelling properties

Fig. 3(A) shows the gel contents of the resulting hydrogels at various total irradiation doses. The gel content data shows that there are enhancements in gel content with the addition of total irradiation doses. A higher dose produces in a higher conversion, and as a result, a higher gel content in the hydrogel can be reached. The gel content increases with the increase in the total irradiation dose [41]. It is well-known that gel content and swelling ratio can also be correlated with the crosslinking density. It means that the greater the gel content, the higher the crosslinking density. Similar results with this report were obtained in hydrogel synthesis using the γ-radiation method from poly(acrylic acid) and poly(ethylene glycol)diacrylate loaded with Ca^{2+} [42] and gelatin/poly (γ-glutamic acid) [43]. A study found that chitosan crosslinked pNIPAAm hydrogels exhibited an increase in grafting percentage and grafting efficiency with the increase in the dose due to the increasing concentration of free radicals formed in the polymer substrate [44].

At pH 7, the swelling ratio decreases significantly with an increase in temperature from 30 °C to 37 °C, and then the swelling ratio is constant from a temperature of 37 °C to 43 °C (Fig. 3(B)). It means that the hydrogels resulting from this research have the

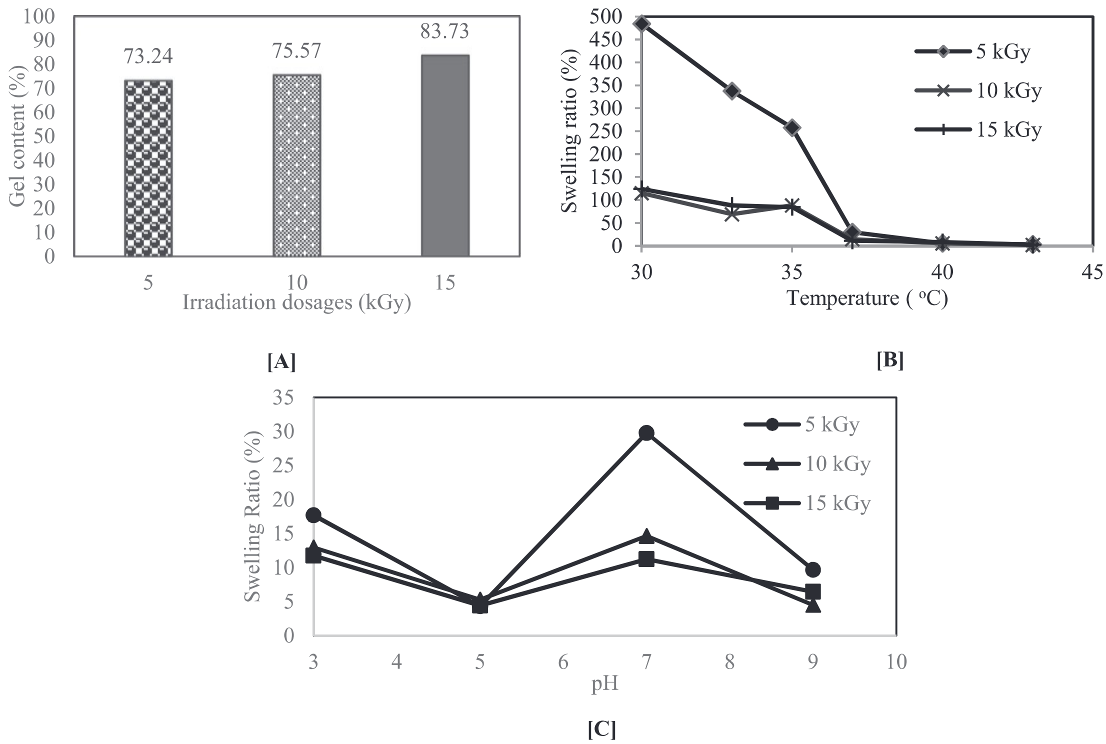

Fig. 3. Physical characteristics of the irradiated hydrogels: [A] gel content, [B] swelling study at different temperatures (pH 7), [C] swelling study at different pHs (temperature 30 °C).

LCST (Lower Critical Solution Temperature) value of 37 °C. The pNIPAAm has hydrophilic and hydrophobic groups affecting unique swelling and deswelling behaviour. The hydrogel swells at a temperature below the LCST and de-swells at a temperature above the LCST. The amount of hydrophilic groups (—NH) and hydrophobic groups (alkyl) in pNIPAAm influences the value of LCST. Hydrophilic groups are commonly weak, so the bonds are easily broken and crosslinked with suitable groups from other materials. The increase in irradiation dose causes a decrease in swelling capacity at various pH conditions. More crosslinked bonds formed due to the irradiation process results in some difficulties for water or aqueous solution to be absorbed into the hydrogel matrix. The swelling behaviour of the hydrogel at different pHs is shown in Fig. 3(C). The highest swelling ratio is reached at pH 7, while de-swelling occurs at pH 9. Unique swelling properties at different pHs are affected by the hydroxyl group (—OH) and amine group (—NH_2) in chitosan [45].

3.3. SEM analysis

In this study, SEM analysis is carried out on hydrogels which have the highest swelling ratio values at various temperatures and pH, namely temperature 30 °C and pH 7. SEM test results for the hydrogels prepared by freeze-thawing followed by irradiation at various magnifications are shown in Fig. 4.

Fig. 4 shows the hydrogel morphology and pore size during the swelling test at 30 °C and pH 7. It can be seen from the Fig. 4 that the hydrogel has relatively uniform pores with an average diameter of 14.6 μm. The results of the SEM analysis show the large hydrogel pore size because at 30 °C and pH 7 the hydrogel experiences swelling with the greatest ratio compared to other temperatures and pHs, and as a consequence, there is a change in the network structure. Changes in the structure of the hydrogel network will result in changes in the diameter size after swelling conditions.

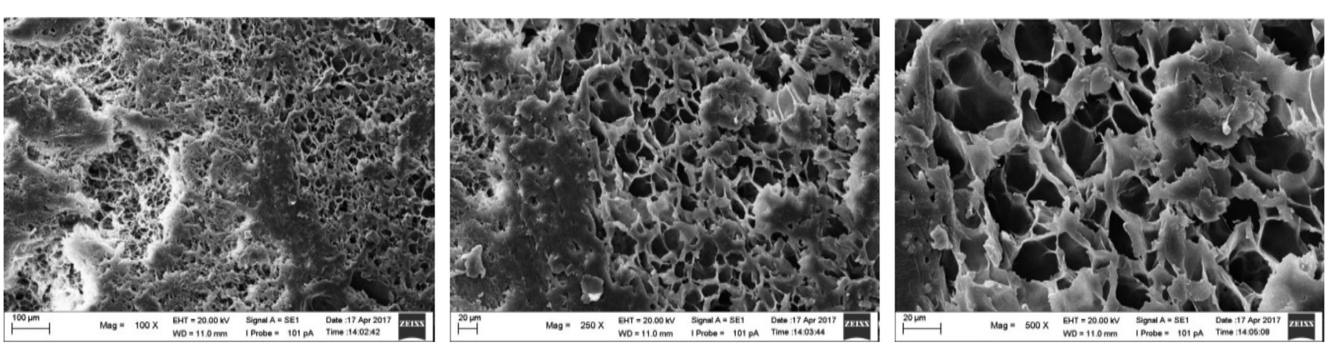

Fig. 4. Irradiated hydrogel SEM image after swelling condition at a temperature of 30 °C and pH 7.

A previous study [27] stated that the method of hydrogel synthesis by freeze-thawing and freeze-thawing followed by irradiation produced larger pores compared to irradiation and irradiation followed by freeze-thawing, but resulted in a smaller swelling value. The pore size is influenced by the hydrogel preparation method in the initial step, but the large pore size does not guarantee the large swelling ability of the hydrogel. The fact is that this current research is carried out using the freeze-thawing stage followed by irradiation and produces the greatest swelling at a temperature of 30 °C and a pH of 7 compared to other temperatures and pHs (Fig. 3). The obtained results need to be compared with previous studies to find a correlation between pore size and the swelling ability of the hydrogel. Based on the results of the swelling test, it indicates that the hydrogel has an LCST of 37 °C at pH 7. At 30 °C, the pore size of the hydrogel will be larger than at 37 °C and 43 °C. This is because at 30 °C and pH 7 the hydrogel has not yet been at LCST temperature so the pores and diameter size of the hydrogel have not undergone a contraction or shrinking process. This is confirmed by a statement that pNIPAAm-based polymer excretes its fluids at a temperature close to that of the human body ~ 37 °C [46].

4. Conclusion

In this work, pH and thermoresponsive hydrogels are successfully synthesized from chitosan, pNIPAAm, and PVA using freezing and thawing methods followed by the γ-irradiation method. The hydrogel with the highest value of gel content (83.73 %) is obtained by the use of 0.5 g of pNIPAAm with a total radiation dose of 15 kGy. The hydrogel with the highest swelling ratio (484.146 %) is obtained by the use of 0.5 g of pNIPAAm with a total radiation dose of 5 kGy at a temperature of 30 °C and a pH of 7. The increase in total radiation dose increases the gel content, and decreases the swelling ratio, but does not give a significant effect on the LCST value. This work confirms that the LCST of the hydrogel is correlated to the pH condition.

CRediT authorship contribution statement

Dhena Ria Barleany: Conceptualization, Writing – original draft, Validation. **Jayanudin:** Supervision, Project administration. **Andriano Suryawan Utama:** Investigation. **Ukas Riyupi:** Resources. **Hafid Alwan:** Data curation, Software. **Retno Sulistyo Dhamar Lestari:** Visualization. **Alia Badra Pitaloka:** Formal analysis. **Meri Yulvianti:** Writing – review & editing. **Erizal:** Methodology.

Data availability

Data will be made available on request.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Jayanudin reports financial support was provided by the Ministry of Education, Culture, Research, and Technology of the Republic of Indonesia.

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Pekanbaru, October 17, 2022

Paper ID: ICChEAS_2022_ paper_9666

LETTER OF ACCEPTANCE

Dear Dhena Ria Barleany

Congratulation, after the pre-peer-review process, we are pleased to inform you that your paper titled "Synthesis and Characterization of Chitosan/Polyvinyl Alcohol Crosslinked Poly(N-Isopropylacrylamide) Smart Hydrogels Via γ-Radiation" has been accepted by the organizer committee of the 3rd International Conference on Chemical Engineering and Applied Sciences (ICChEAS 2022) to be presented at the Conference. The conference will be held online from November 9, 2022. Please kindly find the reviewer's comments in the attachment, you should revise the manuscript based on the comments. Please note, that this Letter of Acceptance is also considered an Invitation Letter. We will appreciate if you make a payment before October 30, 2022. Send your proof of payment to iccheas2022@eng.unri.ac.id. The guideline of payment can be access through this link: http://che.ft.unri.ac.id/iccheas#registration

The proceeding submission portal will be opened on November 25, 2022. Editorial Manager System of Material Today will be used to handle the submission and review process of the manuscript. Please note, that this LoA is not guaranteed that your paper will be published in the Materials Today Proceeding. Therefore, you have to follow the revision process, submission schedule, and author guidelines. For the details of the template and submission process, please visit our website conference. We will be left behind for late submission. Your collaboration will be very helpful to the publishing proceeding.

We are looking forward to welcoming you to our conference

Yours Faithfully,
Chairman of The Committee

ICChEAS

Prof. Amun Amri, ST., MT., Ph.D.

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ICChEAS 2022 submission 9666

1 pesan

ICChEAS 2022 <iccheas2022@easychair.org> 19 Oktober 2022 11.32

Kepada: Dhena Ria Barleany <ria.barleany@untirta.ac.id>

Dear authors,

After the pre-peer-review process, we are pleased to inform you that your paper entitled

Synthesis and Characterization of Chitosan/Polyvinyl Alcohol Crosslinked Poly(N-Isopropylacrylamide) Smart Hydrogels Via γ-Radiation.

has been accepted by the organizer committee of the 3rd International Conference on Chemical Engineering and Applied Sciences (ICChEAS 2022) to be presented at the Conference. The conference will be held online on November 9th, 2022. Please note, that this Letter of Acceptance is also considered an Invitation Letter.
The comments of the reviewer of your manuscript are included at the foot of this letter.
We are looking forward to welcoming you to our conference

Best regards,
Amun Amri.

SUBMISSION: 9666
TITLE: Synthesis and Characterization of Chitosan/Polyvinyl Alcohol Crosslinked Poly(N-Isopropylacrylamide) Smart Hydrogels Via γ-Radiation

---------------------- REVIEW 1 --------------------
SUBMISSION: 9666
TITLE: Synthesis and Characterization of Chitosan/Polyvinyl Alcohol Crosslinked Poly(N-Isopropylacrylamide) Smart Hydrogels Via γ-Radiation
AUTHORS: Dhena Ria Barleany, Jayanudin Jayanudin, Retno Sulistyo Dhamar Lestari, Alia Badra Pitaloka and Erizal Erizal

----------- Overall evaluation -----------
SCORE: 2 (accept)
----- TEXT:
This article can be accepted by double-checking the English grammar and abbreviations that are displayed for the first time must be accompanied by their abbreviations

---------------------- REVIEW 2 --------------------
SUBMISSION: 9666
TITLE: Synthesis and Characterization of Chitosan/Polyvinyl Alcohol Crosslinked Poly(N-Isopropylacrylamide) Smart Hydrogels Via γ-Radiation
AUTHORS: Dhena Ria Barleany, Jayanudin Jayanudin, Retno Sulistyo Dhamar Lestari, Alia Badra Pitaloka and Erizal Erizal

----------- Overall evaluation -----------
SCORE: 3 (strong accept)
----- TEXT:
This paper contains an aspect of novelty and is well written.

9666 LoA Dhena Ria Barleany.pdf
87K

ICChEAS
The 3rd International Conference on Chemical Engineering and Applied Sciences

CERTIFICATE OF APPRECIATION

No: 034/ICChEAS-III/2022

This Certificate Recognizes with Appreciation

Dhena Ria Barleany

For contributions as a Presenter
In the 3rd International Conference on Chemical Engineering and Applied Sciences (ICChEAS)
November 9th 2022, Pekanbaru, Indonesia

ICChEAS

Prof. AMUN AMRI, ST., MT., Ph.D
Chairman of the Committee

TECHNICAL PROGRAM

Wednesday, November 9th 2022-CONFERENCE

Time	Activities
07.30 – 08.00	Virtual Registration
08.00 – 08.10	Opening
08.10 – 08.30	Indonesian Traditional Dance Performance Singing Indonesian National Anthem Du'a Recitation
08.30 – 08.40	Welcome Speech by ICChEAS 2022 Chairman **Prof. Amun Amri, S.T., M.T., Ph.D.**
08.40 – 08.50	Welcoming Speech by Dean of Engineering Faculty **Prof. Dr. Eng. Ir. Azridjal Aziz, S.T., M.T., IPU**
08.50 – 09.05	Opening Speech by Rector of Universitas Riau **Prof. Dr. Ir. Aras Mulyadi, M.Sc., DEA**
09.05 – 09.10	Photo Session (Virtual and Offline)
09.10 – 09.20	Session Break
09.20 – 09.25	Transition from MC to Moderator Moderator Plenary Session I: **Dr.-Ing. Amizon Azizan**
09.25 – 10.30 10.30 – 10.40	Keynote Speech Session I: 1. **Prof. Dr. Chun-Chen Yang** 2. **Prof. Dr. Ing. Ir. Misri Gozan, M.Tech.** Q&A Session I
10.40 – 10.45	Transition from Moderator Plenary Session I to Session II Moderator Plenary Session II: **Idral Amri, S.T., M.T., Ph.D.**
10.45 – 11.45 11.45 – 11.55	Keynote Speech Session II: 1. **Prof. Yazid Bindar, Ph.D.** 2. **Prof. Datuk Dr. Ahmad Fauzi Ismail** **(Presented by Dr. Hasrinah Hasbullah)** Q&A Session II
11.55 – 12.00	Transition from Moderator II to MC
12.00 – 13.30	Lunch Break
13.30 – 17.00	Parallel Session + Co–Host Meeting
17.00	Virtual Closing ceremony by Head of Chemical Engineering Department, Universitas Riau **(Idral Amri, S.T., M.T., Ph.D.)**

PARALLEL SESSION

Chemical Engineering and Applied Chemistry (CEA)
Break Out Room 1, Session 1 (13.30 – 15.00)
Moderator: Zuqni Meldha S.T., M.T.

No	ID Author	Title	Author
1	242	Numerical Investigation of Photonic Crystal Fiber-based Plasmonic Material for Alcohol Concentration Detection	Dedi Irawan, Khaikal Ramadhan, Saktioto, Azhar, Azwir Marwin
2	2528	Deplete the Saturated Fatty Acid Fraction from Palm Biodiesel Fuel with $AgNO_3$ Solvent	Zuchra Helwani, Godlief Fredrik Neonufa, Graecia Lugito, Tirto Prakoso, Rinaldi Idroes
3	411	Lithium-Ion Battery Performance Improvement Using Two-Dimensional Materials	Edy Riyanto, Tony Kristiantoro, Erie Martides, Dedi, Budi Prawara, Dadang Mulyadi, Suprapto
4	1390	The Temperature of The Heat Exchanger Network in the Cleaning Scheduling	Hairul Huda, Tantra Diwa Larasati, Muhammad Rizky Nur Kholik, Tasya Futry Sabilla, Muhammad Faisal Akbar
5	1620	Homogeneity Analysis of B30 Mixing Results with Additives in Mixing Tanks Using Computational Fluid Dynamics (CFD)	Feri Karuana, Hafizh Ghazidin, Suyatno, Andrias R Wimada, Muhammad P Helios, Himawan Sutriyanto, Maharani D Sholikhah
6	5469	The Use of Fractional Factorial Design for Analyzing Effect of Sulfuric Acid Concentration and Temperature on Furfural Yield	Said Zul Amraini, Rozanna Sri Irianti, Nirwana, Sri Rezeki Muria, Novia Liana Sari, Rachmad Aidil Azhar, Reno Susanto

Chemical Engineering and Applied Chemistry (CEA)
Break Out Room 1, Session 2 (15.30 – 17.00)
Moderator: Prof Zuchra Helwani ST., MT., Ph.D.

No	ID Author	Title	Author
1	9666	Synthesis and Characterization of Chitosan/Polyvinyl Alcohol Crosslinked Poly(N-Isopropylacrylamide) Smart Hydrogels Via γ-Radiation	Dhena Ria Barleany, Jayanudin, Andriano Suryawan Utama, Ukas Riyupi, Hafid Alwan, Retno Sulistyo Dhamar Lestari, Alia Badra Pitaloka, Erizal
2	9492	Production of Hydrogen Gas (H_2) and Sodium Hypochlorite (NaOCl) From Seawater Using Photovoltaic-Electrolysis Method	Lukman Hakim, Meriatna, Ishak, A. Setiawan, R. Sari, Fajar
3	2005	Hydrogen Production from Seawater Using H_2SO_4 Catalyst by Photovoltaic-Electrolysis Method	Lukman Hakim, Ratna Sari, Fadli, Fajar, Safrizal
4	2637	Hydrogel Derived from Water Hyacinth and Banana Skin Pectin as a Membrane Layer	Muthia Elmaa, Ni Kadek Devi Ananda Saraswati, Paskah Fransiska Afrida Simatupang, Retno Febriyanti, Aulia Rahma, Fitri Ria Mustalifah
5	3485	Hydrolysis Process of Oil Palm Empty Fruit Bunches for Bioethanol Production with *Saccharomyces cerevisiae*	Adrianto Ahmad, Chairul, Nervi Rita, Riska Wulandari, Vini Alvia Sari
6	3657	Response Surface Methodology for Optimization of Liquid Smoke Production Yield from Durian rinds (*Durio zibethinus Murr.*)	Muhammad Faisal, Suraiya Kamaruzzaman, Hera Desvita, Dini Annisa, Cut Zahara

Chemical Engineering and Applied Chemistry (CEA)
Break Out Room 2, Session 1 (13.30 – 15.00)
Moderator: Prof. Dr. Ir. Nasrul Arahman, M.T.

No	ID Author	Title	Author
1	8690	Response Surface Methodology (RSM) for Corrosion Control by G*igantochloa apus L.* Leaf Extract Toward Low Carbon Steel in Hydrochloride Acid Solution	Komalasari, Abdullah Agung Hayyuka, Syelvia Putri Utami, Evelyn, Ahmad Fadli, Muhammad Iwan Fermi, Desi Heltina
2	6160	Recent Technology of Edible Coating Production: A Review	Vika Andriani, Noer Abyor Handayani
3	602	Effect of Non-Vacuumed and Vacuumed Preparation During Electron Beam Irradiation Wet-Soaked Pretreatment on Oil Palm Crystallinity Index	Amizon Azizan, Nur Amira Aida Jusri, Muhammad Kamarulariffin Mohammed Faisal, Mohd Faizal Abd Rahman
4	1357	Dissolving Pulp from Areca catechu L Fiber by Prehydrolysis, Soda-Cooking and Clorine Free Bleaching Processes	Yusnimar, Al Fikri, Robi Juandry, Drastinawati
5	4311	Economic Evaluation and Sensitivity Analysis of Methanol Plant from Glycerol	Meilani Kusuma Wati, Elmi Sunarya, Nada Zafirah, Hari Rionaldo, Zulfansyah
6	4348	Optimization of the Utilization of Tofu Waste (Whey) and Gypsum ($CaSO_4\,2H_2O$) as Coagulant Material in the Process of Making Tofu from Aceh Soybeans	Jakfar, Azwar, Husni Husin, Abral Muslim, Nadiatul Hikmah

Chemical Engineering and Applied Chemistry (CEA)
Break Out Room 2, Session 2 (15.30 – 17.00)
Moderator: Hermawan Dwi Ariyanto, Ph.D.

No	ID Author	Title	Author
1	6600	Kinetics Modelling of The Solid-Liquid Extraction Process of Linamarin Compounds from Cassava Leaves Assisted By UV-Photobioextractor	Mohamad Endy Yulianto, Retno Dwi Nyamiati, Mega Mustikaningrum
2	6651	Optimization and Characterization of Nypa Fruit Fruit Shell Activated Carbon Irradiated by Microwave in Term of Carbonization Temperatures and Activated Time	Sofia Anita, T. Abu Hanifah, Itnawita, Ganis Fia Kartika
3	8461	Moisture Removal, Colour Changes and Elemental Analysis of *Momordica Charantia* During Far Infrared Drying	Hanafiah Zainal Abidin, Habsah Alwi, Nadzirah Fisol
4	9624	Production of Dissolving Pulp from Gerunggang (Cratoxylum arborescens) Wood Through Pre-hydrolysis and Kraft-SAQ Cooking Process	Chairul, Evelyn, Deviona, Anisa Mutamima, Yeni Aprianis, Drastinawati, Muhammad Dion Arfi, Sendra Erfa Satria, Muhammad Humam Ridho
5	843	Regeneration of Spent Activated Carbon from an Oleochemical Industry Via Microwave Irradiation Technique	Abdul Qahar Mazelan, Siti Shawalliah Idris, Syazana Mohamad Pauzi, Harumi Veny
6	1745	Effect of Torefaction Temperature and Adhesive Amount on The Characteristics of Waste Briquettes Coffee Grounds as An Alternative Renewable Fuel	Novita Aida, Nurhidayatul Fadila, Budiyono, Slamet Handoko

Chemical Engineering and Applied Chemistry (CEA)

Break Out Room 3, Session 1 (13.30 – 15.00)

Moderator: Dr. Eng. Vita Paramita, S.T., M.M., M.Eng.

No	ID Author	Title	Author
1	2380	Extraction of Silica from Rice Husk Ash: Effect of Alkaline and Alkaline Concentration	Sri Aprilia, Cut Meurah Rosnelly, Zuhra, Emir Haffiz Akbar, Muhammad Raqib, Khairul Rahmah, Fitriani, and Amri Amin
2	8762	Investigation of The Non-Isothermal Decomposition Kinetics and Biofuel Properties of Leucaena Leucocephala Pellets Via Torrefaction	Sharmeela Matali, Norazah Abd Rahman, Siti Shawalliah Idris
3	6968	Plastic and Organic Waste Identification Using Multispectral Imaging	Minarni Shiddiq, Dodi Syofyan Arief, Zulfansyah, Khusnul Fatimah, Dilham Wahyudi, Dewi Anjarwati Mahmudah, Dinda Kamia Evka Putri, Ikhsan Rahman Husein, Sinta Afria Ningsih
4	5105	The Effect of Sodium Hydroxide on Delignification, Bleaching on Acid Pretreatment, and Hydrolysis (Sulphuric Acid and Phosphoric Acid) on Glucose Production from Young Coconut Husk	Sri Rezeki Muria, Zikir Akbar Kemala, Abdul Hafiz Hidayat, Rozanna Sri Irianti
5	1056	Formulation and Physical Properties of Citronella Oil Emulsion on Differences in Emulsifiers with the Addition of Maltodextrin	Anggun Puspitarini Siswanto, Hermawan Dwi Ariyanto, Mohamad Endy Yulianto, Mirza Muhammad Faisal, Oktaviani Kusuma Wardani and Dmitriy Kuvshinov
6	7315	Pineapple Leaf Extract as Corrosion Controller for ASTM A36 Steel	Syelvia Putri Utami, Viona Aulia Rahmi, Ahmad Fadli, Khairat, Desi Heltina, Evelyn, Komalasari
7	6434	Experimental Study on Drying Characteristic of Black Tea Using Agitated Vibro Fluidized Bed Dryer	Sri Utami Handayani, Eflita Yohana, Mohammad Tauviqirrahman

Catalysis and Catalytic Reaction Engineering (CRR)

Break Out Room 3, Session 2 (15.30 – 17.00)

Moderator: Idral Amri, S.T., M.T., Ph.D.

No	ID Author	Title	Author
1	4758	Green Solvent on Microwaved Assisted Extraction of Star Fruit	Vita Paramita, Aisya Rohmatul Ummah, Heny Kusumayanti, Rizka Amalia, Wahyu Widyati
2	8241	Catalytic Cracking of Pyrolysis Coconut Shell Oil into Benzene Toluene Xylene with CaO/HZSM-5 Catalyst	Setiadi, Jelita Helianisa
3	1446	Pyrolysis of Citronella Oil Residue Impregnated with Metals (Na & Ni) Into Bio-oil and Char Products and Various Characterization Test	Vira Annisa Indriani, Angelina Grace, Setiadi
4	3783	Glycerolysis of stearic acid using green catalyst	Farra Aisha, Ida Zahrina, Sunarno
5	9749	Utilization of Sugarcane Bagasse into Bio Asphalt Through Pyrolysis Process using Zeolite-Based Catalyst	Heny Dewajani, Windi Zamrudy, Zakijah Irfin, Diana Ningtyas, Noufi Mujibur Ridlo

Biotechnology, Biochemical, Environmental Engineering (BIO)
Break Out Room 4, Session 1 (13.30 – 15.00)
Moderator: Prof. Dr. Ir. Yunardi, MA.Sc.

No	ID Author	Title	Author
1	464	Efficacy of Natural and Fullwashed Post-Harvest Processing Variations on Arabica Coffee Characteristics	Prayoga Bagus Widodo, Mohamad Endy Yulianto, Hermawan Dwi Ariyanto, Vita Paramita
2	1025	Rational Use of Water Resources During the Oil Industry	Maya Abdullayeva and Shams Alizadeh
3	1836	Increased Performance Warning Radiation Security Drinking Mineral the Water of The Territory of The Republic of Azerbaijan	Maya Abdullayeva and Aysun Baxshaliyeva
4	7139	Different types of starch on the characterization and quality of edible film as functional packaging in fresh meat or meat products: A review	Tindy Rahmadi Putri, Alfiana Adhitasari, Vita Paramita, Mohamad Endy Yulianto, Hermawan Dwi Ariyanto
5	9626	Utilization of Lamtoro Fruit Peel Waste to Improve the Performance of Supercapacitor Electrodes in Energy Storage	Erman Taer, Inri Br Pasaribu, Novi Yanti, Apriwandi, Rika Taslim
6	5013	Evaluation of Biomass Residues Combustion Characteristics in Open Burning	Maulana G. Nugraha, Elsava Derangga Mozasurya, Muslikhin Hidayat, Harwin Saptoadi
7	554	Combination of red ginger (Zingiber officinale r.) and banana skin (Musa paradisiaca) as instant powder drink that are rich in antioxidants	Nur Ramadani Fitri and Anggun Puspitarini Siswanto

Biotechnology, Biochemical, Environmental Engineering (BIO)
Break Out Room 4, Session 2 (15.30 – 17.00)
Moderator: Sri Rezeki Muria, S.T., M.P., M.Sc.

No	ID Author	Title	Author
1	7379	Rotary Algae Biofilm Reactor (RABR) Using Microalgae Chlorella sp. for Tofu Wastewater Treatment	Fakhriyah Hanifa Mazaya Nasution, Shinta Elystia, Aryo Sasmita
2	208	Gamma Irradiation Study on Rice Straw to Obtain Sugar	Noor Anis Kundari, Panji Pamungkas Jati, Harum Azizah Darojati, Kartini Megasari
3	1803	Water quality analysis using WQI (Water quality index): A review	Monika Verma, Kuldeep Srivastava, Joshan Gajurel, Nirdesh Regmi Avinandan, Mandesh Kumar Yadav, Sonam Lekzin
4	5128	Removal of Total Dissolved Solids from Oil-Field Produced Water Using Ceramic Adsorbents Integrated with Reverse Osmosis	Netty Herawati, Subriyer Nasir, Muhammad Hatta Dahlan, Maulana Yusuf, Maulid M. Iqbal, Kiagus Ahmad Roni
5	8537	Physically Activated Patchouli Dregs Carbon as A Biosorbent for Remotion of Methylene Blue	Fairuza Hysna, Mariana, Farid Mulana, Mahidin, Syawaliah Muchtar
6	6158	Hesperidin Production from Lime peel (*Citrus aurantifolia S*) using Microwave Assisted Extraction (MAE)	Mohamad Endy Julianto, Alihsan Rahmawati, Aisyah Nuraini, Azizah Azhar, Fatma Sekar Putri Dewanty

Biotechnology, Biochemical, Environmental Engineering (BIO)
Break Out Room 5, Session 1 (13.30 – 15.00)
Moderator: Prof. Dr. Sri Aprilia, M.T., Ph.D.

No	ID Author	Title	Author
1	1298	Hydrophilic Metal-chelated Membrane for Biocatalytic Membrane Reactor Application	Nur Ummi Anisa Muhammad Rasidi, Fauziah Marpani, Nur Hashimah Alias, Nur Hidayati Othman, Muhammad Shafiq Mat Shayuti
2	5106	Palm Fruit Fiber Hydrolysis Process in Fermentation by *Saccharomyces cerevisiae* for Bioethanol Production	Zulfansyah, Adrianto Ahmad, Erika Puji Hartanti, Muhammad Shaza
3	699	Novel Spent Bleaching Earth Industrial Waste as Low-Cost Ceramic Membranes Material: Elaboration and Characterization	Aulia Rahma, Muthia Elmaa, Muhammad Roil Bilad, Isnasyauqiah, Abdul Rahman Wahid, Muhammad Sirajul Huda, Dwi Resa Lamandau
4	8069	Effect of Physical and Biological Pretreatment on Sugarcane Bagasse Waste-Based Biogas Production	Siswo Sumardiono, Hashfi Hawali Abdul Matin, Ihdina Sulistianingtias, Tri Yulianto Nugroho, and Budiyono Budiyono
5	4271	The Abundace of Microplastics in Siak Tributary Sediments in The Watershed Area, Pekanbaru City, Riau (Case Study Air Hitam River and Sago River)	Gunadi Priyambada, Budhi Kurniawan, Rillian Gerry, Lita Darmayanti
6	6869	The Utilization of Silica Sand and Clay with The Addition of Sawdust as Raw Material for Manufacturing Ceramic Membrane to Reduce TSS and TDS Levels of Peat Water	Zuqni Meldha, Idral Amri, Muhammad Dandy Tito Angkoso, Yosia Jumaga

Biotechnology, Biochemical, Environmental Engineering (BIO)
Break Out Room 5, Session 2 (15.30 – 17.00)
Moderator: Assoc. Prof. Ch.M. Dr. Ying Chin Lim

No	ID Author	Title	Author
1	6899	Microwave-based Antioxidant Extraction from Pineapple Peel Waste	Nurhanis Syafiqah Harith, Norazah Abd Rahman, Norashikin Ahmad Zamanhuri, Syafiza Abd Hashib
2	7695	Effect of Electric Voltage and Number of Aluminum Electrodes on Palm Oil Industry Liquid Waste Treatment with Continuous Electrocoagulation Process	Idral amri, Zuqni meldha, Syamsu Herman, Della Karmila, Mhd. Fadilah Ramadani
3	2273	Effect of Addition of Palm Oil Shell Biochar on Carbon Dioxide Emissions in Topsoil	Aryo Sasmita, Ulfa Septianda, Shinta Elystia
4	3903	Microwave Drying Characteristics and Quality of Ananas comosus Peel, Core and Pulp	Nurul Asyikin Md Zaki, Habsah Alwi, Syafiza Abd Hashib, Ummi Kalthum Ibrahim, Junaidah Jai, Norashikin Mat Zain, Nurul Hidayah Samsulrizal
5	3336	PLA/PVDF Film as An Alcohol Sensor for Tapai	Suzihaque Maqsood-ul-Haque, Syarah Syamimi Mazlan, Yang Lu
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4	9220	Production of Bioactive Compounds from *Bacillus paramycoides* LBKURCC218 Co-cultivation	Zona Octarya, Titania T. Nugroho, Yuana Nurulita, Nabella Suraya, Saryono
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ICChEAS PROGRAM BOOK

NOVEMBER 9th
2022
PEKANBARU, INDONESIA

The 3rd International Conference on Chemical Engineering and Applied Science

"Towards the green and sustainable process engineering and applied science technology"

Program Book of The 3rd International Conference on Chemical Engineering and Applied Sciences 2022 (ICChEAS 2022)

Pekanbaru, Riau, Indonesia

November, 9th 2022

Organized by:

Chemical Engineering Department
Universitas Riau, Indonesia

Supported by:

Universitas Syiah Kuala, Indonesia
Vocational School of UNDIP, Indonesia
Universiti Teknologi MARA, Malaysia

WELCOME MESSAGE FROM RECTOR OF UNIVERSITAS RIAU

Assalamu'alaikum warahmatullahi wabarakatuh

In the stead of Allah, Most Generous, Most Merciful. May Allah's mercy, peace, and blessings be upon you. I would like to sincerely thank and welcome everyone to the third International Conference on Chemical Engineering and Applied Sciences on behalf of the Universitas Riau (ICChEAS 2022). The keynote addresses at ICChEAS 2022 will be delivered by Prof. Dr. Chun-Chen Yang, Ming-Chi University of Technology, Taiwan; Prof. Datuk Dr. Ahmad Fauzi, Universiti Teknologi Malaysia; Prof. Dr. Ing. Ir. Misri Gozan, M.Tech. Universitas Indonesia, Indonesia; and Prof. Yazid Bindar, Ph.D., Institut Teknologi Bandung, Indonesia.

The International Conference on Chemical Engineering and Applied Sciences (ICChEAS) collaboratively organized by the Faculty of Chemical Engineering, Universiti Teknologi MARA (UiTM), Malaysia, and 3 universities in Indonesia, the Vocational School, Universitas Diponegoro (UNDIP), Indonesia, the Chemical Engineering Department of Universitas Riau (UNRI) and the Chemical Engineering Department of Universitas Syiah Kuala (UNSYIAH).

The first ICChEAS was held in Banda Aceh in 2019, the second was in Semarang in 2021, and this year's third ICCheAS is being held in Pekanbaru, Riau. The 3rd ICChEAS's main goal is to offer the most sophisticated and extensive international forum for distributing information about research, development, and applications in the fields of chemical engineering and applied science. "Towards Green and Sustainable Process Engineering and Applied Science Technology" is the subject of the third ICChEAS in 2022.

I believe that ICChEAS 2022 will give participants the chance to exchange information, form partnerships and collaborations, and share research progress between universities, institutions, governments, and small-to-large industries, also it be able to benefit society and the country.

I would like to convey my sincere gratitude to the keynote speakers, all of the seminar participants, and the organizing committee for their tremendous efforts in helping to organize the third ICChEAS in 2022. Hopefully, we can all significantly contribute more to the nation's advancement. I would like to extend a sincere appreciation and a warm welcome to all of our esteemed guests and participants.

Wassalamu'alaikum wr wb.

Prof. Dr. Ir. Aras Mulyadi, M.Sc., DEA

WELCOME MESSAGE FROM DEAN OF FACULTY OF ENGINEERING UNIVERSITAS RIAU

Assalamu'alaikum warahmatullahi wabarakatuh

In the name of Allah, the Most Beneficent and the Most Merciful. May peace, mercy, and blessings of Allah be upon you. Good morning, dear colleagues, professors, lecturers, researchers, ladies and gentlemen, and good morning to our participants joining us online, from around the world.

On behalf of the Dean of the Faculty of Engineering Universitas Riau, I would like to express my sincere gratitude and welcome you to the 3rd International Conference on Chemical Engineering and Applied Sciences (ICChEAS 2022) with the theme "Towards Green and Sustainable Process Engineering and Applied Science Technology. This ICChEAS 2022 is organized by the Chemical Engineering Department Faculty of Engineering, Universitas Riau collaborated with the Faculty of Chemical Engineering, Universiti Teknologi MARA (UiTM), Malaysia, the Vocational School, Universitas Diponegoro (UNDIP), and the Chemical Engineering Department of Universitas Syiah Kuala (UNSYIAH).

May I thank the all committees of the 3rd International Conference on Chemical Engineering and Applied Sciences (ICChEAS 2022). We wish to extend a warm welcome to fellow delegates from various countries and also online participants joining this conference. We want to extend warm greetings to all those who support this international conference. Moreover, I honorably welcome our keynote speakers: Prof. Dr. Chun-Chen Yang, Ming-Chi University of Technology, Taiwan; Prof. Datuk Dr. Ahmad Fauzi, Universiti Teknologi Malaysia; Prof. Dr. Ing. Ir. Misri Gozan, M.Tech. Universitas Indonesia, Indonesia; and Prof. Yazid Bindar, Ph.D., Institut Teknologi Bandung, Indonesia.

Last but not least, my deepest gratitude goes to the Advisory Board, Organizing Committee, International Scientific Committee, institutions, companies, and volunteers who have directly and indirectly supported the success of this International Conference. The committee has organized a vibrant scientific program and is working hard to present highly respected and internationally notorious speakers to lead it. Although we try our finest to be professional, on behalf of the Universitas Riau, please accept our sincere apologies should their inconveniences occur before, during, or after the event. I wish you a very productive conference with exciting and encouraging discussions and exchange of knowledge so that together we can anticipate a future of groundbreaking knowledge, research, and technology for humanities.

May God bless us all with good health to make this event a successful and enjoyable one!

Best Regards,

Prof. Dr. Eng. Ir. Azridjal Aziz, S.T., M.T., IPU

WELCOME MESSAGE FROM THE CONFERENCE CHAIR OF 3rd ICChEAS 2022

Assalamu'alaikum warahmatullahi wabarakatuh

Good morning, ladies and gentlemen.

First of all, I would like to thank Allah subhanallahu wata'ala for giving us His blessing so that we could hold this activity. Because of His mercy, we can present in this opening event in good condition and health.

The honorable Rector Universitas Riau or the representative, the dean of Engineering faculty, Universitas Riau, the Head of the Chemical Engineering Department Universitas Riau, keynote speakers, reviewers, presenters, committees, and to all of you who are present today.

Here, I am Amun Amri, on behalf of the chairman of this event, I would like to welcome all of you to the 3rd International Conference on Chemical Engineering and Applied Sciences (ICChEAS) 2022.

This conference is organized by the Department of Chemical Engineering, Universitas Riau, Indonesia, in collaboration with the Department of Chemical Engineering, Diponegoro University, Indonesia, the Department of Chemical Engineering, Syiah Kuala University, Indonesia, and the Department of Chemical Engineering, University Teknologi MARA Malaysia.

This year, the topic of conference is “Toward the Green and Sustainable Process Engineering and Applied Science Technology”. With the successful history of the conference series in the recent five years, we are committed to preparing this conference program for fostering vibrant exchanges and dynamic collaborations among the academic and research communities in dealing with a sustainable process engineering and applied science technology around the world. In this conference, about 95 papers will be presented from around the world namely Indonesia, Malaysia, India, China, Azerbaijan, Brunei, Japan, Nepal, Turkey and UK.

The organization of ICChEAS 2022 gets a great support from the conference organizing team members and conference paper reviewers. We would like to sincerely thank all individuals who have rendered their support in every possible way to make this conference a reality. We would also like to express our gratitude to all the paper authors and registered participants for their stimulating academic contributions to the vibrant intellectual exchange at this conference. With the online setting in the conference program, we hope every participant will have a unique ICChEAS 2022 experience for creating new friendships, professional collaborations, and beautiful memories.
Thank you!

Wassalamualaikum warahmatullahi wabarakatuh

Best regard

Prof. Amun Amri, S.T., M.T., Ph.D.

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Time	:	07.30 – 17.00 (UTC +7)
Organizer	:	Chemical Engineering Department, Universitas Riau
Official Language	:	English
Secretariat		Laboratorium Teknologi Produk Fakultas Teknik Universitas Riau Kampus Binawidya, Jl. HR. Soebrantas Km.12,5, Simpang Baru, Panam Pekanbaru, Riau (28293) Indonesia Phone : (+62-761) 567446; 566937 Fax : (+62-761) 567446 Email : iccheas2022@eng.unri.ac.id Web : http://che.ft.unri.ac.id/
Conference Website	:	http://che.ft.unri.ac.id/iccheas
Conference Link	:	**https://us06web.zoom.us/j/89123241164?pwd=OWVncytWY0MwN0xzVGNEc3JieUQ2Zz09**
Meeting ID	:	891 2324 1164
Passcode	:	2022

Paper ID	**ICChEAS_2022_paper_9666**
Title	Synthesis and Characterization of Chitosan/Polyvinyl Alcohol Crosslinked Poly(N-Isopropylacrylamide) Smart Hydrogels Via γ-Radiation
Authors	Dhena Ria Barleany*, Jayanudin, Andriano Suryawan Utama, Ukas Riyupi, Hafid Alwan, Retno Sulistyo Dhamar Lestari, Alia Badra Pitaloka, Erizal
Abstract	Chitosan-based smart hydrogels with stable mechanical and good flexibility have many desirable qualities and broad applications. In this present study, novel smart hydrogels of chitosan crosslinked with poly(N-Isopropylacrylamide) (pNIPAAm) and polyvinyl alcohol (PVA) was synthesized by freezing and thawing procedures and then subjected to γradiation at room temperature. The effect of irradiation dose on the gel fraction and water absorption characters of chitosan-pNIPAAm hydrogels was investigated. In addition, the structure-property behavior of the hydrogels was characterized by FTIR spectroscopy and thermogravimetric analysis (TGA). The experimental results reveal that the hydrogels synthesized with 15 kGy total dose showed higher gel content (83.73%) compared to 5 kGy and 10 kGy, while the appropriate dose of γ-radiation to achieve the highest absorption in water (484,146%) was 5 kGy. The hydrogel characterization test confirmed that cross-linking occurred between pNIPAAm, PVA, and chitosan. Increasing the radiation dose resulted in more cross-links in the hydrogel and resulted in better thermal stability. In this work confirmed that chitosan/PVA cross-linked pNIPAAm hydrogel with dual pH/temperature stimuli-responsiveness hold the promise to be used in various applications
Keywords	Chitosan; Dual-Responsive; Hydrogel; Radiation; Smart Material

Proceedings of The 3rd International Conference on Chemical Engineering and Applied Sciences

Synthesis and Characterization of Chitosan/Polyvinyl Alcohol Crosslinked Poly(N-Isopropylacrylamide) Smart Hydrogels Via γ-Radiation

Dhena Ria Barleany[1*], Jayanudin[1], Andriano Suryawan Utama[1], Ukas Riyupi[1], Hafid Alwan[1], Retno Sulistyo Dhamar Lestari[1], Alia Badra Pitaloka[1], Erizal[2]

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Abstract

Chitosan-based smart hydrogels with stable mechanical and good flexibility have many desirable qualities and broad applications. In this study, novel smart hydrogels of chitosan crosslinked with poly(N-Isopropylacrylamide) (pNIPAAm) and polyvinyl alcohol (PVA) were synthesized through freezing and thawing procedures and then subjected to γ-radiation at room temperature. The effect of irradiation dose on the gel fraction and water absorption characteristics of chitosan-pNIPAAm hydrogels was investigated. In addition, the structure-property behaviour of the hydrogels was characterized using FTIR spectroscopy and thermogravimetric analysis (TGA). The experimental results revealed that the hydrogels synthesized with a 15- kGy total dose showed a higher gel content (83.73%) compared to 5-kGy and 10-kGy total doses, while the appropriate dose of γ-radiation to achieve the highest absorption in water (484.146%) was 5 kGy. The hydrogel characterization test confirmed that cross-linking occurred between pNIPAAm, PVA, and chitosan. Increasing the radiation dose resulted in more cross-links in the hydrogel and resulted in better thermal stability. this study confirmed that chitosan/PVA cross-linked pNIPAAm hydrogel with dual pH/temperature stimuli-responsiveness holds the promise to be used in various applications.

Keywords: Chitosan; Dual-Responsive; Hydrogel; Radiation; Smart Material

1. Introduction

Hydrogels are a unique class of three- dimensional cross-linked polymeric networks having the capability to hold a large fraction of aqueous solvents and biological fluids within their structures [1]. Hydrogels have emerged as smart materials and have shown great potential in many applications because of their features which are responsive to various external stimuli [2]. Hydrogels as stimuli-responsive polymers display a sharp transition in physicochemical characteristics under a small change in environmental conditions, such as pH, temperature, ionic strength, light exposure, magnetic field, electrical field, ultrasound, etc [3]. With an array of triggering mechanisms, these stimuli-responsive hydrogels allow precise control over basic material properties, such as physical structure, porosity, swelling, and modulus [4]. Their ability to change their conformation and properties when stimulated at different conditions causes them to be used prominently in wastewater treatment [5], sustained release of agrochemicals [6], food science [7], and biomedical field [8].

Smart hydrogels prepared from polysaccharides have many advantages because of their attractive characteristics including biocompatibility, biodegradability [9], self-healing, and responsiveness to environmental stimuli [10]. Among them, chitosan is an excellent excipient because it is an abundant and naturally occurring polysaccharide which has numerous amazing properties such as low toxicity, high biocompatibility, desirable biodegradability [11], and antimicrobial activity [12]. Chitosan which is a derivative of chitin, a natural polymer extracted from crab and shrimp shells, exhibits pH sensitivity and polycationic properties. The external and environmental pH affects the swelling and shrinking behaviour of chitosan hydrogel derived from protonation/deprotonation of the primary amine group (NH_2) [13]. Because of the responsive properties, chitosan is to become an advanced biopolymer in the development of smart polymeric delivery systems [3].

Chitosan has hydrophilic (-OH) and hydrophobic (-NH_2) groups which can be easily modified with other natural or synthetic polymers through physically or chemically cross-linked reaction [14] to produce hydrogels with different stimulus responses. In addition to the pH, the temperature is also one of the most exploited stimuli in 3-dimensional networks due to its ease of use and regulation in testing [13], so the dual responsive chitosan-based hydrogel (pH and temperature) has become a tremendous development in recent years [15; 16; 17]. To improve its performance, chitosan can be modified by crosslinking with other moieties/monomers to increase its sensitivity to the pH and temperature [18]. The group of thermoresponsive polymers includes poly(N-isopropylacrylamide) [16; 17; 19), poly(N,N-diethylacrylamide) [20], and poly (N-vinylcaprolactam) [21].

Poly(N-isopropylacrylamide)(pNIPAAm) is a thermoresponsive polymer which is applied in a wide range of promising applications because of its well-defined structure and characteristics especially its temperature sensitivity is close to a lower critical solution temperature (LCST) [22]. Thus, the pH sensitivity of chitosan and the volume phase transition temperature of pNIPAAm have been the interest of various scientists to develop materials for carrier and controlling the release of active substances [16; 23].

A relatively simple and inexpensive technique that does not involve the use of toxic chemical agents and allows simultaneous sterilization of crosslinked polymers is radiation induction [24]. A previous study used radiation as the crosslinking agent to produce graft copolymers [25], and another previous study used the irradiation method to reduce and control polysaccharides' molecular weight distribution to adjust their water solubility and gelling ability [26]. In this work, smart hydrogels were fabricated from chitosan and N-isopropylacrylamide (pNIPAAm) with the addition of polyvinyl alcohol (PVA) by introducing the combination method of freeze-thawing and γ-irradiation to induce cross-linking. The influence of γ-radiation dose on the gel content, the swelling properties, and the hydrogel thermal characteristics was examined in this study.

2. Materials and methods

2.1. Material

Chitosan (industrial grade, deacetylation degree of 85%, Biotech Surindo), poly(N-isopropylacrylamide) (pNIPAAm; Aldrich), polyvinyl alcohol (PVA; Merck), acetic acid (glacial, Merck), pH buffer (3, 5, 9), and distilled water were used throughout the experiment.

2.2. Preparation of pH- and Thermo-responsive Hydrogels

A total of 0.5 g of chitosan was added into 50 ml of 1 % (v/v) acetic acid solution under a stirring condition to form a chitosan solution. Meanwhile, distilled water as much as 100 ml was added into a bottle containing 5 g of PVA, then the bottle was closed and heated in an autoclave at 120^{o}C for 30 min. PVA solution was then brought out from the autoclave and left at room temperature. After that, 10 ml of PVA solution was mixed with 10 ml of chitosan solution. Then, 0.5 g of pNIPAAm (0.5 g) was added to the mixed solution and then followed by a stirring process at room temperature. The final solution as much as 5 ml was taken and kept in 5-ml tubes and continued with 3 cycles of freezing and thawing procedure, whereas each cycle was run for 24 h (12 h for freezing and 12 h for thawing). After the freezing and thawing procedure, the samples were γ-irradiated under a constant rate of irradiation dose (2.5 kGy/min) at room temperature. The total dose of γ-irradiation for each sample was varied to 5, 10, and 15 kGy. After the irradiation process, the physical samples turned into gels. At this stage, the resulting gels are known as hydrogels. Then, the hydrogels were cut into small sizes (approximately 20 mm^2) and characterized.

2.3. Testing and Characterization

2.3.1. Transform Infra-Red (FTIR)

The samples were characterised using FTIR (Shimadzu IR – Prestige 21) to confirm the presence of functional groups of chitosan, pNIPAAm, and PVA in the hydrogels.

2.3.2. Thermo Gravimetric Analysis (TGA)

In this study, the TGA was examined to find the thermal properties of synthesized hydrogels. During the heating process, there was decreasing in the hydrogel weight due to thermal degradation. The thermal characteristics of the hydrogels were measured using DTG-60 TA WS-60 Shimadzu at a temperature in the range of 20 – 600^{o}C.

2.3.3. Swelling study

The swelling behaviour of the non-crosslinked and crosslinked gels was examined by swelling the gels in media at different pHs and temperatures. Pre-weighed dry hydrogels were immersed in pH-5 buffer solutions at a temperature of 30°C for 24 h. The gels were withdrawn from the solutions at different time intervals and their wet weights were determined after the withdrawn gels were absorbed for the first time with filter papers and immediately weighed. The swelling ratio was determined using the equation (1) based on the study reported by Erizal [27].

$$Swelling\ ratio\ (\%) = \frac{w_s}{w_d} \times 100\% \qquad (1)$$

Where swelling ratio also can be named as the water absorption (% wt) of the gels, Wd and Ws are the sample weights in the dry and swollen states, respectively.

2.3.4. Gel Content Measurement

Hydrogel powder as much as 0.01 g (W_0) was packed in the tea bag. Then, the tea bag containing the hydrogel powder was immersed in distilled water for 24 h at room temperature. After that, the tea bag was dried in an oven for 24 h at 60^{o}C and weighed (W_1). Gel content was determined using the equation (2).

$$Gel\ content\ (\%) = \frac{w_1}{w_0} \times 100\% \qquad (2)$$

where w_1 is the dried gel weight (g) and w_o is the initial gel weight (g).

3. Results and discussion

3.1. Structure Analysis

Chemical modifications induced by the irradiation process were analyzed using FTIR. The spectra of nonirradiated and irradiated hydrogels are shown in Figure 1.

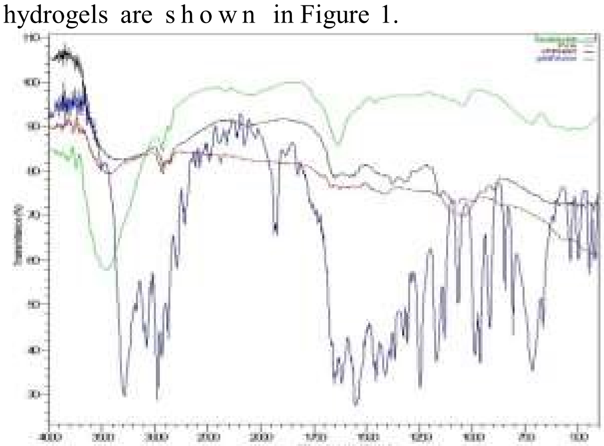

Figure 1. FTIR spectra

In the spectrum of PVA, the peaks were observed at 3402.4 cm^{-1}, corresponding to the O-H groups. The absorbance at 2960.73 cm^{-1} can be attributed to the CH stretching, and the absorbance at 1004.96 cm^{-1} corresponded to the C=O stretching. In the spectrum of pNIPAAm, the absorption bands at 3280.92 cm^{-1}, 2920.35 cm^{-1}, 1546.91 cm^{-1}, and 1170.24 cm^{-1} were attributed to stretching vibration absorption peaks of –NH, C-H, C=O, and C-N, respectively. The spectrum of chitosan showed the peak at 3442.94 cm^{-1} which was attributed to O-H and N-H, the peak at 2920.23 cm^{-1} which corresponded to C-H, and the absorbances at 1589.34 cm^{-1}, 1255.45 cm^{-1}, and 1154.25 cm^{-1} which were attributed to C=O, C-O, and C-N, respectively. When the mixture of chitosan-pNIPAAm-PVA was irradiated, the grafted hydrogel was produced, and its FTIR spectrum was presented in a curved shape. It was found that the spectrum of the hydrogel had the same peaks as the peaks of the spectra of chitosan, pNIPAAm, and PVA, but the intensity of the peak at 3471.87 cm^{-1} was lower compared to that of the same peak in the spectra of chitosan and pNIPAAm. It proved that chitosan, pNIPAAm, and PVA were crosslinked forming an interpenetrating network.

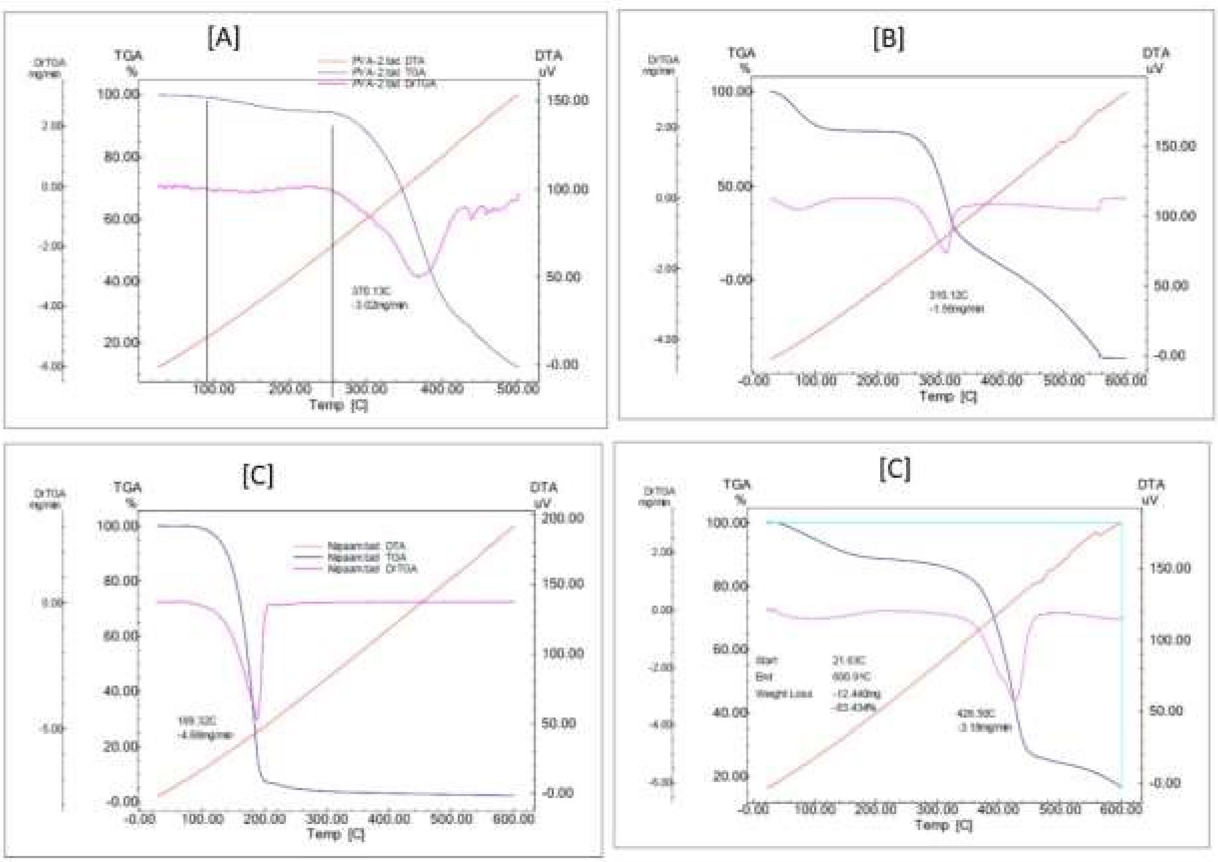

Figure 2 Thermograms of the reactants and the irradiated hydrogel: a) PVA, b) pNIPAAm, c) chitosan, d) irradiated hydrogel

The thermograms presented in Figure 2 show changes in the weight of a compound as a function of temperature and time. The Figure 2 (A) shows the thermogram of PVA. The PVA as much as 10 mg was heated at the temperature range of 20°C to 500°C for 2 h. In the beginning, the PVA sample showed small weight loss due to water evaporation at ±100-200°C, and then the sample weight was continuously degraded. Significant weight loss of PVA was performed at a temperature of 260°C until 500°C. The melting point of PVA was 200°C. Based on TGA (DTG-60) analysis, the biggest value of PVA weight loss was -3.02 mg/min which was obtained at a temperature of 370.13°C. Furthermore, the Figure 2(B) shows the thermogram of pNIPAAm. The pNIPAAm as much as 10 mg was heated from 25°C to 600°C for 115 min. At the heating temperature range of ±100-200°C, there was a decrease in the weight of pNIPAAm. Based on TGA (DTG-60) analysis, the biggest pNIPAAm weight degradation occurred at 189.32°C with a degradation rate of -4.66 mg/min. The Figure 2(C) shows the thermogram of chitosan. The chitosan as much as 10 mg was heated from 25°C to 600 °C for 115 min. Water evaporation occurred in the chitosan sample at a heating temperature of ±30-100°C. The significant decrease in chitosan weight was performed at 260°C and the weight started to be constant at 560°C. Based on TGA (DTG-60) analysis, the biggest value of chitosan weight loss was -1.56 mg/min which was obtained at 310.12°C. Moreover, after the thermal process, the weight of hydrogel resulting from γ-irradiation dose of 5 kGy decreased by -83.434 %, as shown in Figure 2 (D). That value represented the amount of hydrogels that were degraded as a result of heat treatment. The high value of the hydrogel weight loss was correlated to the impurities contained in the samples and the crosslinking homogeneity of the polymer. The radiation caused a rapid crosslinking and affected the increase in the polymer thermal stability.

3.2. Effect of irradiation dose on the gel contents and swelling properties

Figure 3 reports the gel contents and swelling capacities of resulting hydrogels at various total doses of irradiation.

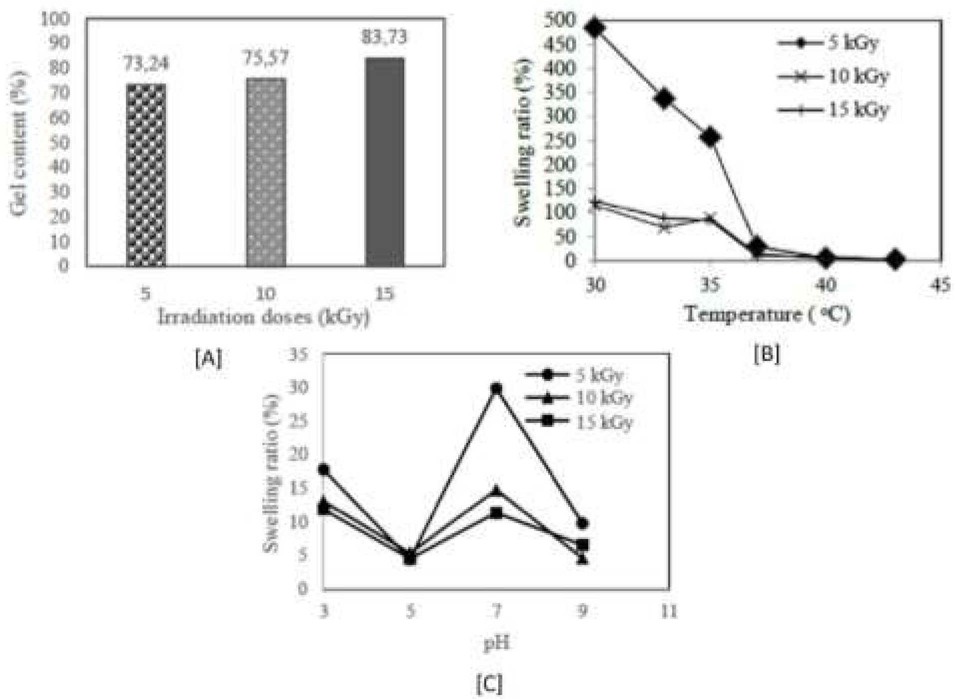

Figure 3. Gel content [A] and swelling study ([B] and [C]) of the irradiated hydrogels

Figure 3(A) shows the gel contents and swelling capacities of the resulting hydrogels at various total irradiation doses. The gel content data showed that there were enhancements in gel content with the addition of total irradiation doses. A higher dose resulted in a higher conversion, and as a result, a higher gel content in the hydrogel can be reached. The gel content increased with the increase in the total irradiation dose [28]. At pH 7, the swelling ratio decreased significantly with an increase in temperature from 30 °C to 37 °C, and then the swelling ratio was constant from a temperature of 37°C to 43 °C (Figure 3(B)). It means that the hydrogels resulting from this research had the LCST (Lower Critical Solution Temperature) value of 37°C. The pNIPAAm has hydrophilic and hydrophobic groups affecting unique swelling and deswelling behaviour. The hydrogel swells at a temperature below the LCST and de-swells at a temperature above the LCST. The amount of hydrophilic groups (-NH) and hydrophobic groups (alkyl) in pNIPAAm influenced the value of LCST. Hydrophilic groups were commonly weak, so the bonds were easily broken and crosslinked with suitable groups from other materials. The increase in irradiation dose caused a decrease in swelling capacity at various pH conditions. More crosslinked bonds formed due to the irradiation process resulted in some difficulties for water or aqueous solution to be absorbed into the hydrogel matrix. The swelling behaviour of the hydrogel at different pHs is shown in Figure 3(C). The highest swelling ratio was reached at pH 7, while de-swelling occurred at pH 9. Unique swelling properties at different pHs were affected by the hydroxyl group (-OH) and amine group ($-NH_2$) in chitosan [29]

4. Conclusion

In this work, pH and thermoresponsive hydrogels were successfully synthesized from chitosan, pNIPAAm, and PVA using freezing and thawing followed by the γ-irradiation method. The hydrogel with the highest value of gel content (83.73 %) was obtained by the use of 0.5 g of pNIPAAm with the total radiation dose of 15 kGy. The hydrogel with the highest swelling ratio (484.146 %) was obtained by the use of 0.5 g of pNIPAAm with the total radiation dose of 5 kGy at a temperature of 30°C and a pH 7. The increase in total radiation dose increased the gel content, and decreased the swelling ratio, but did not give a significant effect on the LCST value. This work confirmed that the LCST of the hydrogel was correlated to the pH condition.

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