International Journal of Current Science Research and Review
ISSN: 2581-8341
Volume 06 Issue 12 December 2023
DOI: 10.47191/ijcsrr/V6-i12-35, Impact Factor: 6.789
IJCSRR @ 2023

Synthesis and Characterization of Nanoherbal of Reed Root Ethanol Extract (*Imperata cylindrica* L) Using Ionic Gelation Method

Shabihisma Auliya Nurdin[1], Suyatno Sutoyo[2]
[1,2] Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Negeri Surabaya, Jl. Ketintang Surabaya, Indonesia

ABSTRACT: The purpose of this study was to determine the characteristics of nanoherbal of reed root ethanol extract (*Imperata cylindrica* L) and optimal variations of alginate and $CaCl_2$ for the manufacture of nanoherbal of reed root ethanol extract. Nanoherbal are made using the ionic gelation method by mixing ethanol extract of reed root, alginate solution, and $CaCl_2$ solution. There are three variations in the composition of alginate and $CaCl_2$ solutions used, namely the ratio of 1: 6.66, 1: 3.33 and 1: 2.5. The solids in the nanoherbal colloids are separated using centrifuges. The residue is washed with aqueous and *freeze dryer* for 24 hours. Colloidal nanoherbal were characterized using PSA to determine particle size and potential zeta values. Nanoherbal solids were characterized using SEM to determine their surface morphological shape. The nanoherbal that were successfully made were faded yellow and white solids. The IR spectrum of nanoherbal shows absorption bands at O-H extended vibrations, C-O bending vibrations, and C=C bending vibrations that undergo shifting, as well as the emergence of manuronat fingerprint bond vibrations in the infrared spectrum supporting the formation of nanoherbal. Characterization using PSA showed a size of 186.2 nm at an optimal concentration ratio of alginic acid and $CaCl_2$ of 1:6.66. SEM results show solids particles are not spherical, uneven, and in the form of loose aggregates on the surface.

KEYWORDS: Ionic gelation method, Nanoherbal, reed root, reed root ethanol extract.

INTRODUCTION

Indonesia has long known and used medicinal plants as one of the efforts to overcome health problems. Knowledge of medicinal plants is based on experience and skills that have been passed down from one generation to the next [1], [2]. One of the plants that has potential as a medicinal plant is reeds (*Imperata cylindrica* L).

Reeds are plants that are considered to interfere with agriculture because they have a bad influence on other plants in getting nutrients, light, and water. However, reeds have benefits for treating vomiting blood, nosebleeds, acute kidney inflammation, and gonorrhea [4], [3]. The efficacy of reeds is reviewed from pharmacology as anti-inflammatory, antidiuretic, antibacterial, and neuroprotective [3]. According to Aryani *et al.* [5], reeds contain secondary metabolites in the form of tannins, saponins, flavonoids, alkaloids, and terpenoids.

Nanoparticles are defined as dispersions of particulates or solid particles with sizes in the range of 10-1000 nm [6]. Nanoparticles derived from plants or plants are called nanoherbal. In the pharmaceutical field, nanoparticles have advantages such as increasing absorption, increasing the solubility of compounds, penetration of active substances, and drug distribution [7], [8].

In this study, nano herbs derived from reed plant extracts were made using the ionic gelation method. The ionic gelation method utilizes a crosslinking process or *crosslinking*, where there is an electrostatic interaction between groups that have different charges between polymers [9]. The release of a drug can occur before it reaches the target cell as a result of its interaction with fluids in the body. Therefore, it is necessary to have a *crosslinking* to inhibit the degradation of polymer nanoparticles and direct that degradation can occur once the drug reaches the target cell [10]. This method was chosen because it has several advantages, namely easier application, less use of solvents, and the materials needed are easy to obtain [11].

In the ionic gelation method, sodium alginate with calcium chloride (CaCl2) acts as a more stable nanoparticle-producing agent [12]. Alginate is one of the natural polysaccharides made from brown seaweed (*Phaeophyceae*), which is used because alginate is a polymer that is not toxic to the body, biodegradable, and biocompatible [13], [14], [15]. Alginate as a polymer has low solubility, low viscosity, and unstable solution stability. Thus, the use of CaCl2 as a crosslinking agent that has multivalent cations is able to increase the viscosity of alginate solutions, which can increase the ability of alginate as a matrix [29].

International Journal of Current Science Research and Review
ISSN: 2581-8341
Volume 06 Issue 12 December 2023
DOI: 10.47191/ijcsrr/V6-i12-35, Impact Factor: 6.789
IJCSRR @ 2023

Ionic gelation methods using sodium alginate and CaCl2 have been used to synthesize nanoherbal from some plant extracts, *Hibiscus rosa-sinensis* L [11], *Boesenbergia pandurata* [15], *Kaempferia rotunda* [14], *Sauropus androgynus* [12]. Research on the synthesis of nanoherbal ethanol extract of reed root using the ionic gelation method with sodium alginate and CaCl2 has never been reported. This study will report on the synthesis of nano herbs from ethanol extracts of reed root plants using ionic gelation methods and their characterization using Zetasizer Nano ZS, FTIR spectrophotometer, and scanning electron microscopy (SEM).

MATERIALS AND METHODS

A. Materials and Tools

The equipment used in this study is SEM (Scanning Electron Microscopy) (HITACHI FLEXSEM 100), PSA (Particle Size Analyzer) (Microtrac), Zeta Sizer nano (Microtrac), FTIR (IRTracer100-Shimadzu), rotary evaporator (Buchi R-300), freeze dryer, centrifuge, a set of maceration tools, beakers, test tubes, ordinary funnels, erlenmeyer, measuring cups 100 mL and 15 mL, pipette volume 5 mL, filter paper, tissue, spatula, *magnetic stirer* (DLAB MS7-H550-Pro).

The ingredients used in this study were reed root, sodium alginate, 96% technical ethanol, p.a ethanol, $CaCl_2$, aquadest, filter paper.

B. Method

1) Sample Preparation of Reed Roots

Separate the reed roots from other parts of the plant first, and then it was washed using water thoroughly. After that, the roots of the reeds were dried at room temperature. The dried reed roots were mashed using a blender to obtain the dried powder.

2) Reed Root Sample Extraction

Five hundred grams of reed root powder was put into the vessel. Added 2.5 L of 96% ethanol solvent and tightly closed. The soaking process was carried out for 24 hours. The mixture was filtered so as to produce extracts and residues. The extract was concentrated using a rotary evaporator (Buchi R-300) at a temperature of 50^{o}C until a thick extract obtained [16].

3) Nanoherbal Synthesis of Reed Root Ethanol Extract

A thick extract of reed root (1 g) was dissolved in 35 mL of ethanol p.a. Then added, 15 mL equates to a 1000 mL beaker. Then 100 mL of alginate solution and 350 mL of $CaCl_2$ solution were introduced into the mixture, and three variations in the composition of the concentration in alginate solution and $CaCl_2$ ratio (1:6.66); (1:3.33) and (1:2.5). Stirring was carried out with a *magnetic stirrer* for 2 hours. The colloidal nanoparticles of reed root extract were then separated by centrifugation to take dissolved solids at a speed of 8000 rpm for 30 minutes. Solid nanoparticles are washed with aquadest. Then the solids are *freeze dryer* for 24 hours. The colloidal nanoparticles formed were characterized using FTIR to determine their functional groups, PSA to determine particle size, and *nano zeta sizer* to determine potential zeta values. The solids that have been formed were characterized using SEM to determine the surface morphological shape of synthesized nanoherbal [15].

RESULTS AND DISCUSSION

A. *Reed Root Extract*

The extraction process uses the maceration method by immersing simplisia into a solvent and soaking 500 g of reed root simplisia using 2.5 L of 96% ethanol solvent for 24 hours. 96% ethanol was selected as solvent Because it is polar so it can attract secondary metabolite compounds such as flavonoids, phenols, saponins, and tannins. The maceration results are separated into filtrate and residue using a vacuum pump. Maceration will produce a concentration equilibrium between the solution inside the cell and outside the cell. The solvent penetrates the cell wall and enters the cell cavity containing the active substance. The difference in concentration outside the cell and inside the cell causes the active substance to dissolve so that a concentrated solution will be removed [18].

The filtrate obtained from maceration is concentrated using a rotary evaporator at 50^{o}C. As a result, a thick brownish-yellow extract of 82.80 g so that a randement of 16.56% was obtained. The sediment obtained from the sample is very necessary because it determines the amount of extract obtained during the extraction process. In addition, the randement data is related to the active substance in the sample, so that if the number of randement increases, the amount of active substance in the sample also increases [19].

International Journal of Current Science Research and Review
ISSN: 2581-8341
Volume 06 Issue 12 December 2023
DOI: 10.47191/ijcsrr/V6-i12-35, Impact Factor: 6.789
IJCSRR @ 2023

B. ***Synthesis of Reed Root Ethanol Extract***

In this study, nanoherbal of reed root ethanol extract were synthesized using the ionic gelation method prepared from alginate and $CaCl_2$ with three variations in $CaCl_2$ concentration. The variation in $CaCl_2$ concentration used is 0.04%, 0.03%, and 0.02%, with the amount of thick extract of reed root as much as 1 g and alginate 0.1 g. Nanoherbal synthesized reed root extract in the form of faded yellow colloidal, as seen in Figure 1.

Figure 1. Formula for Synthesis of Nanoherbal from Reed Root Ethanol Extract

C. ***Size Characterization of Nanoherbal***

Based on characterization testing using PSA, the three formulas have shown a size that corresponds to the vulnerable nanoparticle size, which is <1000. However, the optimal formula that has the smallest nanoparticle size is a sample with a ratio of alginate and $CaCl_2$ (1: 6.66) with alginate 0.1% and $CaCl_2$ 0.02% of 186.2 nm. The formula is used as the optimal formula because if the particle size is small, then the surface area is larger, so the release of the drug will be faster [20]. The size of nanoparticles also determines how easily they enter cells. The smaller the particle size, the easier it is to enter the cell and the greater its absorption in the body [21].

Table 1. Nanoherbal particle size of reed root ethanol extract

Sampel	Alginat (%)	$CaCl_2$ (%)	Ukuraan Partikel (nm)	Nilai Indeks Polidispersitas (PI)
F1	0.1	0.04	210.70	0.1845
F2	0.1	0.03	198.44	0.5610
F3	0.1	0.02	186.20	0.2523

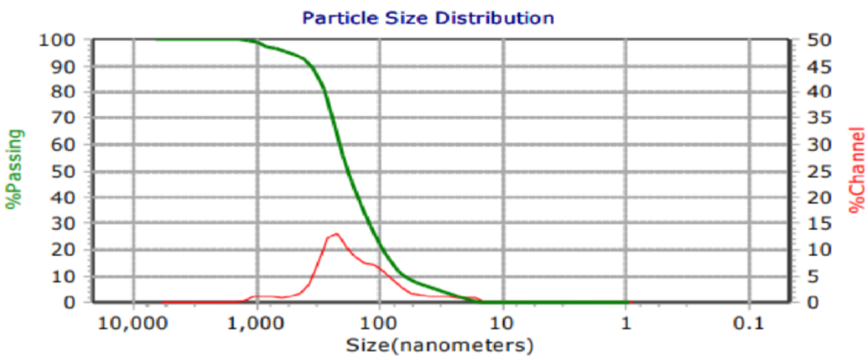

Figure 2. PSA Measurement Result of Nanoherbal F3

In addition to particle size, the polydispersity index (PI) value is also used as a parameter that indicates the particle size distribution of nanoparticles during the analysis process. The PI value ranges between 0 and 1. A PI value close to zero indicates that the

International Journal of Current Science Research and Review
ISSN: 2581-8341
Volume 06 Issue 12 December 2023
DOI: 10.47191/ijcsrr/V6-i12-35, Impact Factor: 6.789
IJCSRR @ 2023

nanoparticle has a homogeneous particle size distribution. In contrast, a PI value of more than 0.5 indicates that the nanoparticle distribution is very heterogeneous or inhomogeneous [22][23].

Table 1 showed that the polydispersity index values of F1 and F3 were less than 0.5. This can mean that the resulting nanoparticle formulation has a homogeneous or uniform particle size distribution and a tendency to be physically stable so as to prevent the particles from aggregating [22][24]. If there is a tendency for nanoparticles to aggregate, it will cause the nanoparticle diameter size to be non-uniform. Aggregation can alter the reactivity and interaction of nanoparticles with an organism. Aggregation of nanoparticles can also change their chemical and physical characteristics and reduce the surface area ratio [25].

D. Characterization of Nanoherbal Functional Groups

FTIR spectra (Figure 3) showed that peak wavenumber in alginate has shifted from 3452.64 cm^{-1} to 3424.67 cm^{-1}, which is characteristic of O-H extended vibrations that indicate the presence of hydroxyl groups in nanoherbal. The vibrational shift of hydroxyl groups (O-H) in nanoherbal is due to the interaction between hydroxyl groups in alginate and hydroxyl groups of phenolic compounds contained in ethanol extract of reed roots. Shifts also occurred at wavenumber 1246.04 cm^{-1} nanoherbal, which indicated the stretching vibration of the C-O bond, and absorption at wavenumber 1616.38 cm^{-1}, which showed the vibration of the C=C bond compared to ethanol extract of reed roots. Changes in the intensity of uptake in nanoherbal with reed root ethanol extract can provide clues to functional groups that play a role in the formation of nanoherbal, namely O-H, C-O, and C = C groups.

In addition, the emergence of the absorption region of wavenumber 810–850 cm^{-1} in the nanoherbal spectrum indicates the presence of vibrations of manuronate fingerprint bonding which is a typical region of alginate. This is a clue that nanoherbal already interact with alginate. Wavenumbers 1616.38 cm^{-1} and 1411.92 cm^{-1} also appear in the nanoherbal spectrum, indicating the presence of asymmetric COO- and symmetrical COO- groups, which are specific regions of alginate.

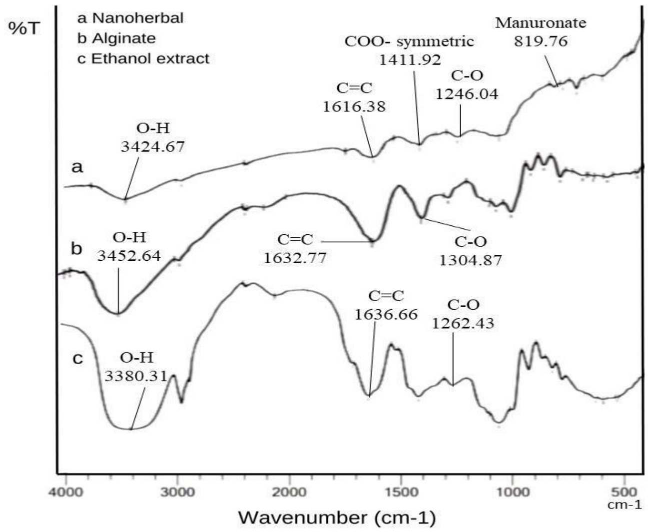

Figure 3. FTIR Spectrum of Nanoherbal (a), Alginate (b), and Ethanol Extract (c)

E. Surface Morphology Characterization of Nanoherbal

SEM analysis in this study was carried out with magnifications of 5000x, 10,000x, and 20,000x. The results of characterization are seen from the optimum formula resulting from particle size characterization and particle distribution so the formula used is F3. SEM of nanoherbal F3 showed a spherical (spherical), uneven, and loosely aggregated surface on the surface. If the nanoparticle preparation is not spherical, it will easily aggregate when in contact with other nanoparticles [26][27] while on an uneven surface caused by an imperfect cross-linking reaction [28][29].

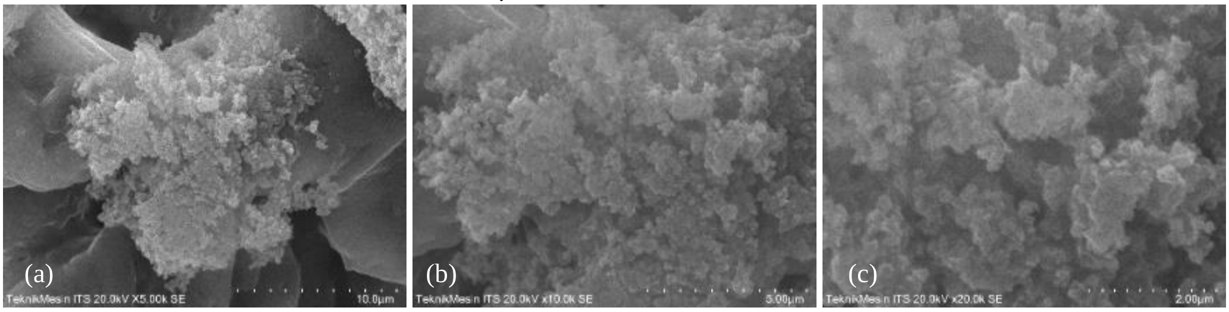

Figure 6. SEM Nanoherbal Reed Root Ethanol Extract (a) 5,000x magnification, (b) 10,000x magnification, and (c) 20,000x magnification

CONCLUSION

Based on the research that has been done, it can be concluded that reed root extract can be made by ionic gelation method using alginate and $CaCl_2$ with an optimal ratio of 1: 6.66. The IR spectrum of nanoherbal shows absorption bands at O-H extended vibrations, C-O bending vibrations, and C=C shifting bending vibrations, as well as the emergence of manuronate fingerprint bond vibrations supporting the formation of nanoherbal in the F3 formula. The synthesized nanoherbal have a particle size of 186.2 nm. SEM results show that nanoherbal is spherical, uneven, and in the form of loose aggregates on the surface.

REFERENCES

1. Bustanussalam. 2016. Utilization of Traditional (Herbal) Medicine as Alternative Medicine. *BioTrends*, *7*(1), 20–25.
2. Wahyuni, N. P. S. 2021. Implementation of Traditional Medicine in Indonesia. *Jurnal Yoga dan Kesehatan*, *4*(2), 149–162.
3. Mulyadi, M., Wuryanti, W., & Sarjono, P. R. 2017. Minimum Inhibition Concentration (KHM) of Reed Sample Rate (*Imperata cylindrica*) in Ethanol Through the Disc Diffusion Method. *Jurnal Kimia Sains Dan Aplikasi*, *20*(3), 130–135.
4. Hairiah, K., van Noordwijk, M., & Purnomosidhi, P. 2000. *Reclamation of Imperata grassland using agroforestry*. Bogor: Internasional Centre for Research in Agroforestry.
5. Aryani, P., Kusdiyantini, E., & Suprihadi, A. 2020. Isolation of Alang-alang Leaf Endophytic Bacteria (*Imperata cylindrica*) and their Secondary Metabolites with Potential as Antibacterials. *Jurnal Akademika Biologi*, *9*(2), 20–28.
6. Mohanraj, V. J., & Chen, Y. 2006. Nanoparticles-A Review. *Tropical Journal of Pharmaceutical Research*, *5*(1), 561–573.
7. Rismana, E., Kusumaningrum, S., Idah, R., Nizar, & Yulianti, E. 2013. Stability Testing of Antiacne Preparations Made from Active Raw Materials Chitosan Nanoparticles / Mangosteen Extract - *Centella asiatica*. *Buletin Penelitian Kesehatan*, *41*(4), 207–216.
8. Sutoyo, S., Amaria, A., Sanjaya, I. G. M., Hidayah, R., Sari, D. P., Dwitarani, N., Oktavia, F. D., & Fadzlillah, N. A. 2022. Synthesis of Nanoherbal from Ethanol Extract of Indonesian Fern *Selaginella plana* and Antibacterial Activity Assay. *Tropical Journal of Natural Product Research*, *6*(1), 44–49.
9. Mardiyanto, M., Herlina, H., Fithri, N. A., & Rahmi, Y. 2019. Formulation and Evaluation of Submicro Preparations of Gelasic-Ionic Particles Carrying Leaf Extract *Pluchea indica* As an antibacterial on the skin of male white rats of Wistar strains. *Jurnal Sains Farmasi & Klinis*, *6*(2), 171–179.
10. Wulandari, I. O., Rahayu, L. B., Riva'i, I., Sulistyarti, H., & Sabarudin, A. 2021. Synthesis and Characterization of Biocompatible Polymer Modified Fe_3O_4 Nanoparticles and Their Potential as Drug Conductors. *The Indonesian Green Technology Journal*, *10*(1), 1–8.
11. Ariani, L. W., & Purwanto, U. R. E. 2019. Hibiscus Leaf Extract Nanoparticle Formulation (*Hibiscus rosa-sinensis* L.). *Repository STIFAR.*

12. Ngafif, A., Ikasari, E. D., Ariani, & Wahyu, L. 2020. Optimization of The Combination of Sodium Alginate and Calcium Chloride ($CaCl_2$) As a Cross-linking Agent of Katuk Leaf Ethanol Extract Nanoparticles (*Sauropus androgynus* (L.) Merr). *BIMFI*, *2*(1), 13–23.
13. Jayanudin, J., Rochmadi, R., Renaldi, M. K., & Pangihutan, P. 2017. The Effect of Coatings On The Encapsulation Efficiency of Red Ginger Oleoresin. *ALCHEMY Jurnal Penelitian Kimia*, *13*(2), 275–287.
14. Khakim, A. N., & Atun, S. 2017. Manufacture of Pepet Key Extract Nanoparticles (*Kaempferia rotunda*) with Alginates at Various Variations in Calcium Ion Concentrations. *Jurnal Kimia Dasar*, *6*(1), 43–51.
15. Putri, G. M., & Atun, S. 2017. Making Characterization of Key Ethanol Extract Nanoparticles (*Boesenbergia panduration*) On Various Variations In Alginate Composition. *Jurnal Kimia Dasar*, *6*(1), 19–25.
16. Puspitasari, A. D., & Proyogo, L. S. 2017. Comparison of Maceration and Socleation Extraction Methods Against Total Flavonoid Levels of Kersen Leaf Ethanol Extract (*Muntingia calabura*). *Jurnal Ilmiah Cendekia Eksakta*, *13*(2), 1–8.
17. Swarbrick, J. 2007. *Encyclopedia of Pharmaceutical Technology Third Edition* (J. Swarbrick (ed.); Third, Vol. 1). New Yok: Informa Heath Care.
18. Sukeksi, L., Sidabutar, A. J., & Sitorus, C. 2017. Alkaline Maceration of Batan Banana (*Musa paradisiaca*) Using Aquadest Solvent. *Teknik Kimia USU*, *6*(4), 22–28.
19. Hasnaeni, Wisdawati, & Usma, S. 2019). The Effect of The Extraction Method On The Yield and Phenolic Content of BetaBeta Wood Plant Extract (*Lunasia amara* Blanco). (*Jurnal Farmasi Galenika (Galenika Journal of Pharmacy) (e-Journal)*, *5*(2), 166–174.
20. Maharani, P., Ikasari, E. D., Purwanto, U. R. E., & Bagiana, I. K. 2022. Optimization of Na-Alginate and Ca-Chloride On Nanoparticles of Fucoidan Purified Extract From Brown Seaweed (*Sargassum polycystum*). *Jurnal Farmasi Medica/Pharmacy Medical Journal (PMJ)*, *5*(2), 38–45.
21. Imanto, T., Prasetiawan, R., & Wikantyasning, E. R. 2019. Formulation and Characterization of Nanoemulgel Preparations of Aloe Vera Powder (*Aloe Vera* L.). *Pharmacon: Jurnal Farmasi Indonesia*, *16*(1), 28–37.
22. Taurina, W., Sari, R., Hafinur, U. C. H., Isnindar, & Wahdaningsih, S. 2017. Optimization of Speed and Stirring Time Against the Size of Chitosan Nanoparticles-70% Ethanol Extract of Siamese Orange Peel (*Citrus nobilis* L. var Microcarpa). *Traditional Medicine Journal*, *22*(1), 16–20.
23. Abdassah, M. 2017. Nanoparticles With Ionic Gelation. *Jurnal Farmaka*, *15*(1), 45–52.
24. Hatmayana, R., Noval, Ramadhani, R. A., Auliyani, N., &; Mardiyah, D. 2022. Characterization of Lemongrass Leaf Extract Nanocapsules (*Chromolaena odorata* L.) With Chitosan-Alginate Variations Using the Emulsion-Diffusion Method. *Jurnal Surya Medika*, *8*(3), 187–194.
25. Masakke, Y., Sulfikar, & Rasyid, M. 2015. Biosynthesis of Silver Particle-Nano Using Mangosteen Leaf Metanol Extract (*Garcinia mangostana* L.). *Jurnal Sainsmat*, *IV*(1), 28–41.
26. Martien, R., Adhyatmika, Irianto, I. D. K., Farida, V., & Sari, D. P. 2012. Development of Nanoparticle Technology As a Drug Delivery System. *Majalah Farmaseutik*, *8*(1), 133–144.
27. Amin, R.R. & Sutoyo, S. 2022. Synthesis and Characterization of Nanoherbal of Reed Root Ethanol Extract (*Imperata cylindrica* L) Using Ionic Gelation Method. *International Journal of Current Science Research and Review*, *5*(7), 2396–2403.
28. Rahman, R.A. & Sutiyo, S. 2022. Synthesis and Characterization of Silver Nanoparticles from Ethanol Extract of Meniran (*Phyllanthus niruri* L.) Using Bioreduction Method. *International Journal of Progressive Science and Technologies*, *34*(2), 32-40.
29. Laksanawati, R., Husni, A., &; Ustadi. 2017. Development of Alginate Extraction Method from Seaweed *Turbinaria ornata*. *Jurnal Pengolahan Hasil Perikanan Indonesia*, *20*(2), 362–369.

Cite this Article: Shabihisma Auliya Nurdin, Suyatno Sutoyo (2023). Synthesis and Characterization of Nanoherbal of Reed Root Ethanol Extract (Imperata cylindrica L) Using Ionic Gelation Method. International Journal of Current Science Research and Review, 6(12), 7791-7796