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Formulation of Encapsulated Extracts from Local Palu Red Onion Bulbs (*Allium cepa* var. aggregatum L.) Using Carrageenan Coating: Characterization, Stability, and Antioxidant Activity

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Abstract. This study aimed to identify the optimal concentration of carrageenan as a coating matrix for extracts of local Palu red onion bulbs (*Allium cepa* var. aggregatum L.) using the ion gelation method to characterise their properties, stability and antioxidant activity. The technique of converting liquid samples into small particles is known as microencapsulation. Carrageenan was formulated at varying concentrations (0.1%, 0.2%, 0.3%) with the addition of 0.1% Na-TPP. Testing of antioxidant activity at a concentration of 0.3% yielded the highest antioxidant activity of 87.59%. The ANOVA test results showed that the matrix concentration had a significant effect ($\rho<0.05$) on antioxidant activity. A total phenolic content of 49.61 mg GAE/g was observed in the 0.3% carrageenan sample. Carrageenan was stable at temperatures of 25°C and 40°C. FTIR characterisation of the encapsulation process indicated the presence of spectra showing interactions between the coating matrix and the extract, revealing changes in functional groups. PSA analysis indicated a particle size of 2100 μm.

Keywords: *Antioxidants, Local red onions from Palu, characterization, encapsulation, stability*

Abstrak. Penelitian ini bertujuan untuk mengidentifikasi konsentrasi karagenan yang optimal sebagai matriks penyalut ekstrak umbi bawang merah lokal Palu (*Allium cepa* var. aggregatum L.) dengan menggunakan metode gelasi ionik untuk memperoleh karakterisasi, stabilitas, dan aktivitas antioksidan. Teknik mengubah sampel cair menjadi partikel kecil disebut mikroenkapsulasi. Karagenan diformulasikan dengan variasi konsentrasi (0,1%, 0,2%, 0,3%) dan penambahan Na-TPP 0,1%. Hasil pengujian aktivitas antioksidan pada konsentrasi 0,3% memperoleh aktivitas antioksidan tertinggi sebesar 87,59%. Hasil uji ANOVA menunjukkan bahwa konsentrasi matriks berpengaruh signifikan ($\rho<0,05$) terhadap aktivitas antioksidan. Total kandungan fenol sebesar 49,61 mg GAE/g ditunjukkan pada karagenan 0,3%. Pada suhu 25°C dan 40°C, karagenan stabil. Karakterisasi enkapsulasi dengan uji FTIR mengindikasikan adanya spektrum yang memperlihatkan interaksi antara matriks penyalut dan ekstrak yang memperlihatkan perubahan pada gugus fungsi. Analisis PSA menunjukkan ukuran partikel 2100 μm.

Kata kunci: *Antioksidan, bawang merah lokal Palu, enkapsulasi, karakterisasi, stabilitas*

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INTRODUCTION

Indonesia possesses extremely abundant biological resources. Plants are one such resource that has been extensively developed into modern medicines. Generally, phytochemical compounds such as phenolics, flavonoids, coumarin derivatives, and others are found in these plants. These substances protect the human body from free radicals. Red onions are one of the plants capable of neutralising free radicals (Juwita *et al*., 2020). Shallots (*Allium cepa* var. aggregatum L.), a better variety in the Palu Valley, are produced

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in the city of Palu. Red onion output in Palu City reached 4,599.00 kw/qui in 2023, according to BPS statistics (2024). This indicates that Central Sulawesi's top horticulture product is red onions (Faidah *et al*., 2020).

According to Martha (2019), bioactive compounds such as quercetin are found in abundance in red onion bulbs (*Allium cepa* L.), which are the richest source of antioxidants. These compounds protect the body from reactive oxygen species caused by external factors or produced by normal oxygen metabolism. The Palu Valley onion (*Allium ascolinicum* L.) exhibited an inhibition value of 67.87 per cent, indicating the presence of very potent antioxidants, according to research conducted by Juwita *et al*. (2020).

Environmental factors including light, oxygen, temperature, and pH might cause polyphenolic chemicals in red onions to degrade more quickly during extraction, formulation, and storage (Hajrin *et al*., 2021). Consequently, a formulation that may increase their efficacy and stability is required.

Encapsulation is one technique that may be utilised to increase the stability and reactivity of bioactive substances. Bioactive substances may be controlled and protected via encapsulation technology, which also has the benefit of efficiently absorbing bioactive molecules through cells and delivering them to specified destinations. Natural polymers including chitosan, carrageenan, pectin, and CMC may be used to create encapsulation (Carboxyl Methyl Cellulose). Among the various types of matrices used for encapsulation, carrageenan, a natural, water-soluble, negatively charged galactose-containing polysaccharide derived from red algae *Rhodophyceae* has been shown to possess exceptional potential due to its biocompatibility, gel-forming ability, and its capacity to create stable microcapsules through thermal or ion-induced gelation (hydrogel formation). When compared with maltodextrin, gum arabic or alginate, carrageenan is capable of producing a stronger gel structure and offers superior moisture-barrier characteristics, which are highly beneficial. The negative charge of carrageenan facilitates electrostatic interactions with peptides, which in turn enhances structural stability and slows down the release process (Vaishnav *et al*., 2025).

Ionic gelation is more often utilised for natural materials, although a number of encapsulating techniques, including spray drying, solvent evaporation, and ionic gelation, have been extensively used to increase the durability and efficacy of active compounds (Sitinjak *et al*., 2025). The ionic gelation process can yield excellent encapsulation efficiency, with rates exceeding 87 per cent for certain formulations, according to research by Erwiyani *et al*. (2024). Ultrasonication is used in the ionic gelation process to reduce the size of polymer molecules (Wahyudi, 2018). Highly stable encapsulation delivery systems can be formed by combining several coating materials with Na-TPP using the ionic gelation process (Windi *et al*., 2022). The encapsulation matrix is reinforced through cross-linking using Na-TPP (Ningsi *et al*., 2014).

Research into the encapsulation of extracts from local Palu red onion bulbs using carrageenan has not yet comprehensively examined the effect of varying carrageenan concentrations on the physicochemical characteristics, stability and antioxidant activity of the extracts, particularly in terms of preserving their characteristics and bioactive compounds during the encapsulation process and storage. The aim of this study was therefore to compare the encapsulation of an extract of the local Palu onion (*Allium cepa* var. aggregatum L.) using carrageenan at various concentrations (0.1%, 0.2% and 0.3%) in order to determine the most optimal concentration for protecting and regulating the release of active compounds.

MATERIAL AND METHODS

Materials

Local Palu red onion bulbs (*Allium cepa* var. aggregatum L.) were among the resources utilised in this investigation. 96% ethanol, DPPH reagent (*2,2-diphenyl-1-picrylhydrazyl*), Na2CO3, distilled water, Folin-Ciocalteu, carrageenan, gallic acid, Na-TPP 0.1%, aluminium foil, filter paper, and tissue paper were additional items used.

Instrumentation

An assortment of tools and instruments were utilised for this investigation, such as a blender, knife, UV-VIS spectrophotometer, ultrasonic device, FTIR, dehydrator, digital balance, Petri dishes, Erlenmeyer flasks, beakers, measuring flasks, cups, droppers, micropipettes, hot plates, magnets, stirring rods, stirrer, oven, microcentrifuge, stopwatch, incubator, glass funnel, PSA, substance spoon, reagent bottle, container (jar), 60 mesh sieve, spray bottle, rack, and test tube.

Procedure

Preparation and production of sample

The shallot bulbs are separated from their roots and skins, washed thoroughly under running water, and then sliced thinly to speed up the drying process. The sliced bulbs are then dried in an oven at 50 ℃. Afterwards, the red onion sample is ground into a powder using a blender and then sieved through a 60-mesh sieve to ensure a uniform particle size. The simplisia is stored in a tightly sealed, dry container in a room protected from direct sunlight.

Extraction method

Using the maceration method, 800 g of dried crude drug powder was weighed out at a ratio of 1:8 and placed in a jar to prepare a 96% ethanol extract from local Palu red onions. It was then stored in a place protected from direct sunlight for 48 hours. The liquid was filtered using a vacuum filter after being stirred periodically every 12 hours during the maceration phase. The resulting filtrate was reconcentrated using a dehydrator at 45 °C after being evaporated using a rotary evaporator (Maryam et al., 2020).

Production of encapsulated extracts from local Palu red onion bulbs

Encapsulation was carried out using the ionic gelation method with carrageenan as the coating agent. Carrageenan was used for encapsulation as it contains various bioactive substances, such as antioxidants, vitamins, and essential oils, thereby enhancing stability and preserving the activity of the bioactive compounds during storage. The process began with the preparation of carrageenan solutions at varying concentrations (0,1 %, 0,2% w/v, and 0.3%) dissolved in distilled water with gentle heating until completely dissolved. Next, 0.3 g of local Palu red onion bulb extract was weighed out and dissolved in 35 mL of 96 per cent ethanol in a beaker. A total of 10 mL of the liquid extract was added to 18 mL of the chitosan solution whilst stirring using a homogeniser at 1500 rpm for 30 minutes. Next, 2 mL of a 0.1% Na-TPP solution was added to the carrageenan solution dropwise whilst stirring using a magnetic stirrer at 1500 rpm for 30 minutes until nanoparticles formed (Windy *et al*., 2022). Sonication was then carried out using a sonicator for 2 hours to break down the polymer molecules into smaller particles (Cahyani *et al*., 2025).

Encapsulation formulation of local palu red onion bulb extract

Table 1. Encapsulation formulation of local Palu shallot bulb extract with varying amounts of carrageenan coating

Formulation	Extract	Carrageenan (% b/v)	Na-TPP (% b/v)
X_0	10 mL	-	-
X_1	10 mL	0.3	0.1
X_2	10 mL	0.2	0.1
X_3	10 mL	0.1	0.1

Antioxidant activity test using the DPPH (2,2-diphenyl-1-picrylhydrazyl) method

According to Boeing *et al*. (2014), to test the antioxidant activity of carrageenan coating formulations at different concentrations, namely 0.1 %, 0.2%, and 0.3% (w/w), 1 mL of sample was drawn up with a micropipette and transferred to a test tube, then 3 mL of a 50 μM *2,2-diphenyl-1-picrylhydrazyl* solution in methanol was added. After the mixture was homogenised and left to stand in the dark for half an hour, the absorbance was measured using a UV-Vis spectrophotometer set at a wavelength of 517 nm. The antioxidant activity of each sample was determined using this method and reported as the percentage of free radical scavenging, calculated using the following formula.

$$\text{Inhibition (\%)} = \frac{Blank\ absorbance - sample\ absorbancel}{Blank\ absorbance} \times 100 \quad ..(1)$$

Total phenol content test (Gultom et al., 2021)

Test tubes were filled with a formulation of carrageenan and an ethanol extract of local Palu red onion bulbs (*Allium cepa* var. aggregatum L.). 1 mL of sample and 5 mL of distilled water were added to each test tube. After 0.5 mL of *Folin-Ciocalteu* reagent had been homogenised, 1.5 mL of 20% Na_2CO_3 was added, and the mixture was incubated for two hours. Gallic acid was used as a standard solution for the determination of total phenolic concentration. The absorbance readings were converted to total phenols and expressed in mg GAE/g sample weight after absorbance was measured at a wavelength of 750 nm.

Stability testing of the extracellular encapsulation of local Palu red onions

This stability test was carried out by storing samples of red onion bulb extract encapsulated using carrageenan at the optimal concentration based on the results of antioxidant activity and total phenolic content. The samples were stored for 24 hours at 4°C, 30°C (room temperature), and 40°C over three cycles. Stability was assessed based on changes in sample turbidity using a turbidimeter and expressed in NTU (*Nephelometric Turbidity Units*) (Cahyani et al., 2025).

Characterization test of local Palu red onion bulb extract

a. Infrared Spectra

FTIR (*Fourier Transform Infrared Spectroscopy*) characterisation was carried out to determine the functional groups in the encapsulation formulation of red onion bulb extract and carrageenan with the addition of Na-TPP. The carrageenan with the optimal concentration, based on the results of antioxidant activity and total phenolic content, was subsequently subjected to FTIR analysis to identify functional groups and interactions, as indicated by shifts in the wavenumbers and intensities of each functional group (Putri *et al.*, 2018).

b. Characterization of PSA

Characterisation was carried out using a PSA (Particle Size Analyser). The carrageenan solution at the optimal concentration, based on antioxidant activity and total phenolic content, was then placed in a cuvette after being mixed with 3 mL of dispersant. The cuvette was subsequently illuminated with visible light, resulting in diffraction. The principle of visible light scattering is used to measure particle size. The wavelength range for maximum scattering is 1–5000 μm (Daskar *et al.*, 2022).

Data Analysis

This study employed a completely randomised design (CRD) with one factor and three replicates. Data on total phenolic content, stability tests, FTIR and PSA were evaluated descriptively, whilst antioxidant activity data were analysed using one-way ANOVA.

RESULT AND DISCUSSION

Extract of Local Palu Red Onion Bulbs

The local Palu red onions (*Allium cepa* var. aggregatum L.) used in this study were collected from a single location in Palu, had their roots and skins removed, and were washed under running water to remove any dirt. The red onions were then sliced thinly to speed up the drying process. The onions were sliced and dried at a temperature of 50°C in an oven. Drying was carried out to reduce the moisture content in the onions and facilitate the extraction of chemical compounds. As excessively high temperatures can alter the chemical structure of bioactive compounds in plants, which would affect the overall flavonoid content, the temperature must not exceed 60°C (Widayanti *et al.*, 2023). To obtain a uniform particle size, the dried samples were ground into a powder using a blender and then sieved through a 60-mesh sieve. Using 96% ethanol as the solvent, 800 g of red onion bulbs were extracted, yielding 217.1 g of concentrated extract with an extraction yield of 27.13%. As flavonoid molecules are polar, ethanol was used as the solvent (Wahyuni *et al.*, 2021).

Antioxidant Activity of Local Palu Red Onion Bulb Extract

The antioxidant activity of an ethanol extract from local Palu shallots was tested using the DPPH (2,2-diphenyl-1-picrylhydrazyl) method, with absorbance measured at 517 nm using a UV-Vis spectrophotometer.

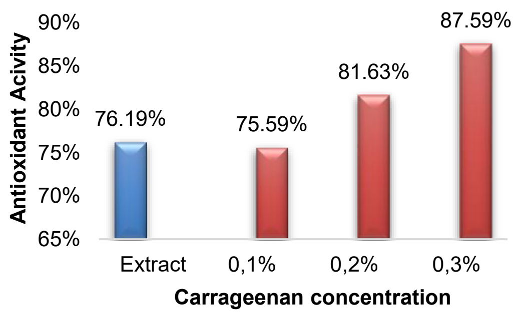

Figure 1. Results of antioxidant activity testing of carrageenan coating formulations

The results of the antioxidant activity test on an extract of local Palu red onions, using an encapsulation technique with a natural carrageenan coating at concentrations of 0.1%, 0.2%, and 0.3% and the addition of 0.1% Na-TPP, are presented in Figure 1. Encapsulation with a carrageenan coating at a concentration of 0.3 per cent yielded the highest free radical scavenging value of 87.59 %, which was higher than that of the pure extract 76.19%, as determined by antioxidant activity measured using the DPPH method. The addition of red onion bulb extract (*Allium cepa* var. aggregatum L.) to the carrageenan coating resulted in a change in the matrix through hydrogen bonding between the phenolic -OH groups and the $-SO_3^-$ and –OH groups present in the carrageenan. Red onion bulbs are rich in flavonoids such as quercetin, whilst carrageenan itself possesses antioxidant properties due to its phenolic compounds derived from hydrocolloid molecules. This leads to an interaction between quercetin and the film matrix that helps maintain its antioxidant properties. The antioxidant activity of quercetin is associated with mechanisms such as chelation of free metals, free radical scavenging, and enzyme modulation (Senggul & Sen, 2025).

According to research conducted by Atmaka *et al*. (2021), carrageenan can protect antioxidant compounds. The more double-helix structures formed by carrageenan, the more effective its protection of phenolic compounds against heat and the prevention of rapid degradation. The results of the ANOVA test showed that the antioxidant activity of encapsulated extracts from local Palu red onion bulbs (*Allium cepa* var. Aggregatum L.) was significantly influenced by the matrix concentration *($p<0.05$)*. This is consistent with research conducted by Fajrin et al. (2025), which showed that the effect of matrix content on the encapsulation efficiency of antioxidant compounds from kecombrang flower petal extract becomes increasingly significant as the matrix content increases.

Total Phenol Content of Local Palu Red Onion Bulb Extract

The *Folin–Ciocalteu* method, which is based on the reaction between phenolic compounds and the *Folin–Ciocalteu* reagent (containing phosphomolybdic acid and phosphotungstic acid) and produces a blue molybdenum-tungstate complex, was used to determine the total phenolic content in this study (Antasionasti *et al*., 2020). The encapsulated formulation coated with 0.3% carrageenan exhibited the best antioxidant activity, based on the antioxidant activity data. Therefore, this formulation was selected for further analysis of total phenolic content, stability, FTIR, and PSA. Figure 2 shows the results of the analysis of the total phenolic content of the encapsulated extract from local Palu red onion bulbs.

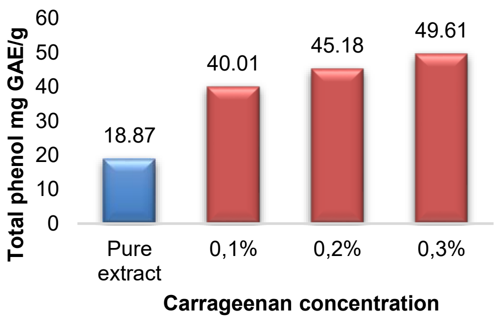

Figure 2. Total phenol content of pure extract and carrageenan formulation

Red onion bulb ethanol extract had a total phenol content of 18.87 mg GAE/g, according to Figure 4.2. The natural carrageenan coating formulations with different concentrations of 0.1%, 0.2%, and 0.3% with the addition of 0.1% Na-TPP produced results of 40.01 mg GAE/g, 45.18 mg GAE/g, and 49.61 mg GAE/g, respectively. The findings demonstrate that after the encapsulation procedure, carrageenan's total phenol concentration rose. The antioxidant activity in fending off free radicals rose in tandem with the rise in carrageenan content. The inclusion of carrageenan can raise the overall phenol content since it includes phenolic compounds that may neutralise free radicals, according to Aripudin *et al*. (2022).

An increase in total phenolic content may indicate better retention of phenolic compounds following encapsulation; however, the possibility of matrix interference with the *Folin–Ciocalteu* method must also be taken into account. The *Folin–Ciocalteu* reagent is not an effective radical scavenger (Senet *et al*., 2018). Research conducted by Mahardani & Yuanita (2021) indicates that total phenolic content does not always correlate directly with antioxidant activity. An increase in total phenolic compounds leads to a corresponding rise in antioxidant activity; conversely, a decrease in total phenolic compounds is not always accompanied by a corresponding decrease in antioxidant activity. This is generally due to the presence of other antioxidant compounds, apart from phenolic compounds, which are more stable during processing or storage.

Stability Test of Encapsulated Extracts from Local Palu Red Onion Bulbs

A sample of the encapsulated preparation at a concentration of 0.3%, which had been shown to be the maximum concentration effective in neutralising free radicals, was used for stability testing. Over three cycles, the encapsulated sample was stored at temperatures of 4°C, 25°C and 40°C. Some natural substances are encapsulated to enhance or maintain their activity, protect them from external influences, and make them more stable and effective during storage (Pratama et al., 2021). Figure 3 shows the results of the stability test for turbidity in the carrageenan layer.

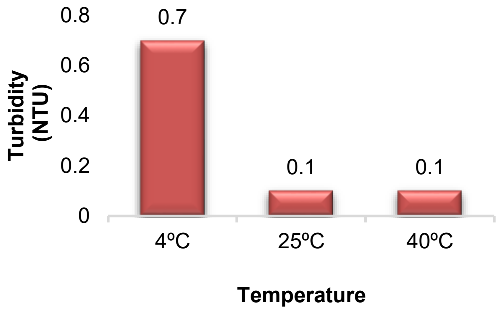

Figure 3. Turbidity value

The results of storage at 4°C, 25°C, and 40°C, as shown in Figure 3 above, indicate the physical stability of the carrageenan layer in relation to aggregate formation. Based on Figure 3 above, the carrageenan layer exhibits physical stability in relation to the formation of large aggregates at 4°C, which causes an increase in turbidity to as high as 0.7 NTU during storage at 4°C, 25°C and 40°C. Carrageenan becomes turbid due to gel formation, which reduces its solubility (Evanuarini & Nidhal, 2024). The carrageenan layer is more chemically and physically stable at 25°C and 40°C. Carrageenan can form a robust matrix capable of preserving and protecting phenolic compounds from environmental degradation, according to research conducted by Vaishnav *et al*. (2025). Carrageenan becomes turbid due to gel formation, which reduces its solubility (Evanuarini & Nidhal, 2024). Carrageenan layers are more chemically and physically stable at temperatures of 25°C and 40°C. Carrageenan can form a robust matrix capable of controlling and protecting phenolic compounds from environmental degradation, according to research conducted by Vaishnav *et al*. (2025). Findings obtained during storage at 4°C, 25°C and 40°C, as shown in Figure 3 above, indicate that the carrageenan layer exhibits physical stability associated with the formation of large aggregates at 4°C, leading to an increase in turbidity of up to 0.7 NTU. Carrageenan becomes turbid due to gel formation, which reduces its solubility (Evanuarini & Nidhal, 2024). Carrageenan films are more chemically and physically stable at 25°C and 40°C. The presence of the 3,6-anhydrogalactose ring ensures that the rigid double-helix structure of the carrageenan chains, or the glycosidic bonds (1-3 and 1-4), remains intact. Carrageenan can provide a robust matrix to preserve and protect phenolic compounds from environmental degradation, according to research by Vaishnav *et al*. (2025).

Characterisation Test of Encapsulation of Local Palu Red Onion Bulb Extract

The functional groups in carrageenan derived from red onion bulb extract and Na-TPP during the encapsulation process of the red onion bulb extract were identified through FTIR characterisation. The wavenumbers and intensities of each functional group, as well as changes in the spectral peaks of specific functional groups indicating the formation of cross-links that reinforce the nanocapsule structure, can be used to describe these interactions.

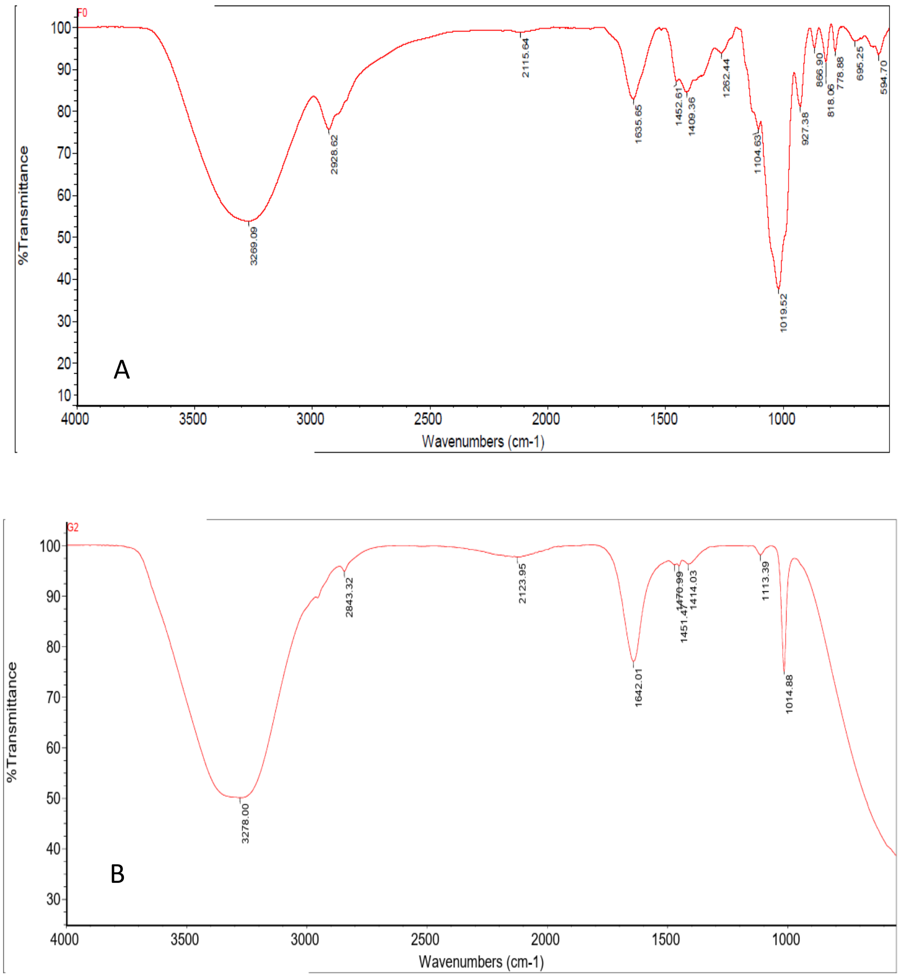

Figure 4. FTIR spectrum of pure extract (A) and 0.3% carrageenan (B)

As shown in Figure 4, the FTIR spectrum of the extract reveals an O–H functional group characteristic of alcohol at a wavelength of 3269.09 cm^{-1}, a C–H functional group indicative of an aliphatic chain at a wavelength of 2928.62 cm^{-1}, the C=O or C=C functional group, indicating an aromatic peak at a wavelength of 163≡5.05 cm^{-1}, the C–O functional group indicating flavonoid glycosidic bonds, and the C–H functional group at wavelengths of 778.88–594.70 cm^{-1}, indicating bending vibrations. This indicates that the functional groups identified are characteristic of red onion bulb extract. When carrageenan was added, the spectral data of the extract showed the characteristic O–H functional group of alcohol at a wavelength of 3728.00 cm^{-1}. This shift in wavelength indicates an interaction between the extract and the carrageenan layer. As flavonoid molecules possess hydroxyl (–OH) groups, this suggests that the encapsulation using a carrageenan layer contains flavonoid compounds (Tapalina *et al*., 2022). Furthermore, the C–H functional group exhibits a shift in wavelength to 2843.32 cm^{-1}, indicating the presence of an aliphatic carbon chain; the C≡C or C≡N functional group at a wavelength of 2123.95 cm^{-1}, indicating the presence of a C=O or C=C functional group at a wavelength of 1642.01 cm^{-1}; CH_2 or CH_3 functional groups at a wavelength of 1451.47–1414.03 cm^{-1}, indicating the presence of an alkyl structure; C–O or C–O–C functional groups at a wavelength of 1113.39 cm^{-1}; and the presence of an alcohol compound at a wavelength of 1014.88 cm^{-1}. The presence of C–O bonds in the 3,6-anhydrogalactose moiety indicates that the active compound has been well encapsulated within the carrageenan matrix, according to Jaya *et al*. (2019). However, these spectral changes cannot yet be used as definitive evidence of the formation of new chemical bonds or *cross-linking* between the extract and carrageenan. Therefore, the FTIR results in this study are more appropriately used as supporting evidence for the occurrence of molecular changes or interactions during the encapsulation process, rather than as sole confirmation of *cross-linking*.

A particle size analyser (PSA) can be used to characterise the encapsulation of red onion bulb extract using a 0.3% carrageenan coating.

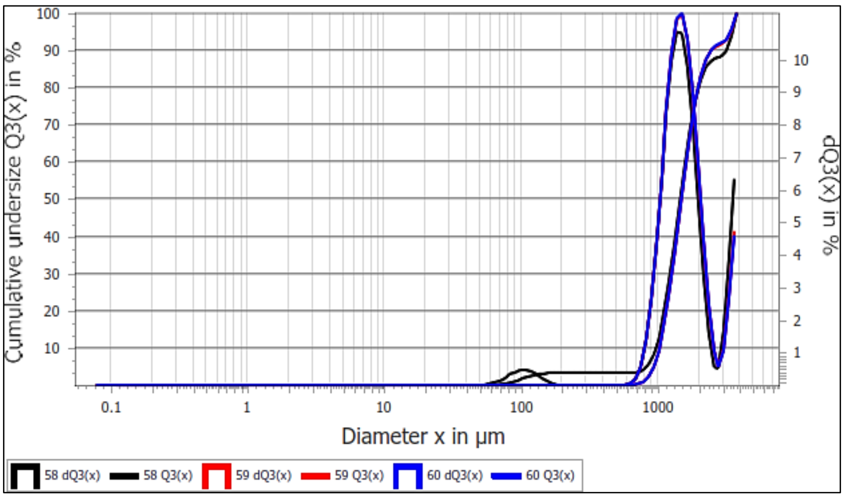

Figure 5. Particle size of encapsulated shallot bulb extract + 0.3% carrageenan

The results of the PSA analysis, as shown in Figure 5 above, indicate that the microencapsulated extract of local Palu red onions, with the addition of Na-TPP, had an average formation rate of 83.75% and a particle size of 2100 μm. These findings are consistent with the requirements for microencapsulation, where particle sizes range from 1-5,000 μm and contain a core substance (Mardikasari *et al*., 2020). Microencapsulation is a process involving the application of a relatively thin coating to small particles of solid substances or liquid droplets and liquid dispersions, which enables controlled release and is relatively straightforward in delivery systems (Sucianti *et al*., 2020).

CONCLUSION

The results of the study indicate that the antioxidant activity of an extract from local Palu red onion bulbs, formulated in encapsulated form, is enhanced. The pure extract exhibited antioxidant activity against free radicals of 76.19%, whilst after encapsulation using a 0.3% carrageenan coating, it demonstrated higher antioxidant activity of 87.59%. The concentration of carrageenan significantly increased the extract's antioxidant activity ($\rho<0.05$). In the total phenolic content test, a concentration of 0.3% yielded the highest total phenolic content of 49.61 mg GAE/g. The product encapsulated with 0.3% carrageenan remained stable at temperatures of 25°C and 40°C. Based on FTIR characterisation, shifts in functional groups indicated the presence of chemical interactions between the extract and the carrageenan layer, whilst the recorded particle size of 2100 μm indicated that the resulting system remained on the micrometre scale.

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