

# GELATIN FROM JERBUNG SHRIMP SHELLS (*Fenneropenaeus marginatus de Man*) USING THREE TYPES OF SOLVENT: ACETIC ACID, PHOSPHORIC ACID, AND SULFURIC ACID

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**Abstract:** Gelatin is a type of protein consisting of amino acids produced through the hydrolysis of collagen. One natural source that can be used as a raw material for making gelatin is the shrimp shell. This study aims to determine the characteristics of gelatin made from Jerbung shrimp shells (*Fenneropenaeus marginatus de Man*) using three types of acid solvents, namely, acetic acid, phosphoric acid, and sulfuric acid. The hydrolysis process by soaking with acid solvent was carried out with varying concentrations of 1%, 3% and 6%. The results of the analysis of the physical characteristics of gelatin with acetic acid, sulfuric acid and phosphoric acid were suitable for SNI. The pH values ranged from 4.5–6.5, the water content was less than 16%, the ash content was 3.25%, the viscosity ranged from 1.5–7 cP, and the gel strength ranged from 50–300 blooms. The FTIR data of gelatin synthesized from 3 types of acids revealed typical absorption peaks, such as N–H and O–H functional groups at wavenumbers of 3600–3400 cm<sup>-1</sup>, C–H groups 3000–2840 cm<sup>-1</sup>, and C=O groups at wavenumbers of 1800–1650 cm<sup>-1</sup>. These results indicate that Jerbung shrimp shells have potential as alternative raw materials for making gelatin.

**Keywords:** Gelatin, Shrimp shell, Acetic acid, Phosphoric acid, Sulfuric acid

**Abstrak:** Gelatin adalah sejenis protein yang terdiri dari asam amino yang dihasilkan melalui hidrolisis kolagen. Salah satu bahan yang dapat digunakan sebagai bahan baku pembuatan gelatin adalah cangkang udang. Penelitian ini bertujuan untuk mengetahui karakteristik gelatin berbahan dasar cangkang udang jerbung (*Fenneropenaeus marginatus de Man*) dengan menggunakan tiga jenis pelarut asam yaitu asam asetat, asam fosfat, dan asam sulfat. Proses hidrolisis dilakukan dengan variasi konsentrasi masing-masing 1%, 3% dan 6%. Hasil analisis sifat fisik gelatin dengan asam asetat, asam sulfat dan asam fosfat menunjukkan hasil yang sesuai dengan persyaratan Standar Nasional Indonesia (SNI). Hasil analisa sesuai dengan standar SNI yaitu nilai pH pada kisaran 4,5-6,5, kadar air di bawah 16%, kadar abu 3,25%, viskositas pada kisaran 1,5-7 cP dan kekuatan gel pada kisaran dari 50-300 bunga. Hasil analisis FTIR gelatin hasil sintesis 3 jenis asam menunjukkan adanya serapan khas gugus

fungsi N-H dan O-H pada bilangan gelombang 3600-3400  $\text{cm}^{-1}$ , gugus C-H pada bilangan gelombang 3000-2840  $\text{cm}^{-1}$ , C= Golongan O pada bilangan gelombang 1800-1650  $\text{cm}^{-1}$ . Hasil tersebut menunjukkan bahwa cangkang udang jerbung mempunyai potensi sebagai bahan baku alternatif pembuatan gelatin.

**Kata kunci:** Gelatin, Cangkang udang, Asam asetat, Asam fosfat, Asam sulfat

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## INTRODUCTION

Indonesia's main increasing production in the field of fishery exports is shrimp. According to the Indonesian Ministry of Trade, shrimp occupy the eighth position of the ten main export products that are relatively easy to trade, resulting in an increase in shrimp export production every year (Islam, Khan & Alam 2016). Shrimp are exported in a frozen state, so that during the freezing process, waste such as the skin of Jerbung shrimp (*Fenneropenaeus marginatus* de Man) is produced (Zhuo et al. 2017).

Jerbung shrimp is a fishery product that is widely developed in the territory of Indonesia from aquatic and aquaculture products (Wiradana et al. 2022). In general, various methods have been used to process fishery products into useful processed products, including products derived from solid waste. With processing technology, fishery products can be utilized and have added value, such as flour processing, shrimp paste processing and processing of chitin and chitosan from the skin or head of shrimp or fish, which

have no economic value (Venugopal 2021). Jerbung shrimp are a type of commercial shrimp that has high economic value. Jerbung shrimp have several advantages for use as a national aquaculture commodity, such as the ability to mature gonads relatively quickly, fast growth rates, and tolerance to changes in salinity (Wiradana et al. 2022).

The increase in shrimp shell waste is still a problem, especially the smell problem that is emitted, which will have an impact on pollution to the environment, and a solution must be found so that the shrimp waste can be utilized and provide added value to the community in shrimp processing production. Shrimp waste produced such as heads, tails and shells, has several main components, such as protein (25%-40%), chitin (15%-20%) and calcium carbonate (45%-50%). The high protein content in the skin can be used as the main ingredient for making gelatin (Said 2020).

Gelatin has benefits in the food industry such as the use of stabilizers, gelling agents and edible coatings.

Moreover, in the cosmetic industry, gelatin is used as an ingredient for making creams (Oktaviani, Perdana & Yus Nasution 2017). Gelatin is a type of protein consisting of amino acids. Gelatin is produced from the acidic, basic processes (Sántiz-Gómez et al. 2019) and enzymatic activity of the collagen network (Hidayat, Nurcahya Dewi & Rianingsih 2016). Generally, the acid process is most widely used because it is relatively easy. The acid process is also preferable because the acid soaking process is relatively shorter than the base process is, and the acid process affects the quality of the gelatin because the basic molecules that form collagen or tropocollagen are denatured by the treatment. For example, acids, such as collagen fibers, cause shrinkage and breakdown of the structure into random coils that dissolve in water, which is called gelatin (Samatra et al. 2022).

The acid process is recommended because acid is able to change triple helix collagen fibers into single chains (Sántiz-Gómez et al. 2019). The type of acid used in the gelatin production process greatly influences the yield, gel strength and viscosity produced. On the basis of the ease of the process of forming gelatin using acid, this research synthesized gelatin using three types of acid. The

gelatin resulting from acid extraction is of good quality (Samatra et al. 2022). Shrimp shell waste has great potential as a raw material for making gelatin with three types of acids. The use of three types of acids is an alternative in gelatin synthesis.

## **METHOD**

### ***Materials***

The materials used in this study were Jerbung shrimp shells taken from PT Bogatama Marinusa (Bomar), aluminum foil, acetic acid ( $\text{CH}_3\text{COOH}$ ), phosphoric acid ( $\text{H}_3\text{PO}_4$ ), and sulfuric acid ( $\text{H}_2\text{SO}_4$ ) with concentrations of 1%, 3% and 6%, respectively, in aquades ( $\text{H}_2\text{O}$ ), ice cubes, calico cloths and water.

### ***Instrumentation***

The tools used were the Shimadzu brand Fourier transform infrared (FTIR-ATR) instrument. FTIR analysis was carried out according to Sulistyani (2018:41) with the FTIR spectroscopy procedure, first setting the reflectance mode (ATR-FTIR) on FTIR spectroscopy. After the reflectance mode is ready, a background scan is performed. Samples of gelatin powder of approximately 0.05 grams were prepared on a sample holder plate. Then, the samples were pressed on the gelatin sample. The test was carried out according to the SOP for the Perkin Elmer IR

Spectrophotometer. Model: Frontier S/N: 96772 in the wavenumber range of 4000  $\text{cm}^{-1}$ , 600  $\text{cm}^{-1}$ , with 10 readings and a resolution of 8  $\text{cm}^{-1}$ . The resulting spectrum was analyzed via the FTIR Software Spectrum Software (Perkin Elmer).

### ***Procedure***

#### ***Sample preparation***

Jerbung shrimp shells were separated from the meat and washed with running water until clean. The clean skin was soaked in warm water for approximately 15 minutes and then dried in the sun for 1–2 days. Then, the next preparation and analysis of gelatin were carried out.

#### ***Synthesis of Gelatin***

Gelatin production was carried out following the procedure of Ait Boulahsen et al. (2018), with slight variation in the procedure, where the shrimp shells were cut to a size of  $\pm 1 \text{ cm}^2$ . Shrimp shells that have been reduced in size are soaked with acetic acid, phosphoric acid, and sulfuric acid at concentrations of 1%, 3% and 6%, respectively. The skin:solvent mixture (1:10 w/v) was incubated for 24 hours and neutralized to a pH close to 7, followed by an extraction step with a skin:water ratio of 1:3 w/v at 70°C for 3 hours. The filtering process was carried out with a

calico cloth to separate the residue, and drying was continued via a freeze dryer for 2 days.

#### ***Total yield***

The total yield was calculated using the Association of Office Analytical Chemist (AOAC) method (1995), where the fresh shrimp shells that had been stored in the refrigerator were then washed with running water. The shrimp shells were then drained and weighed ( $\pm 100$  grams). The yield was calculated as follows:

$$\text{Yield} = \frac{\text{dry weight}}{\text{fresh weight}} \times 100\%$$

#### ***Water content analysis***

Water content analysis was carried out using the Association of Office Analytical Chemist (AOAC) method (1995). The petri dishes were dried and sterilized in an oven at 105°C for 3 hours, cooled in a desiccator for 30 minutes and then weighed. One gram of the mashed sample was weighed in a container with a known constant weight and then dried in an oven at a temperature of 100°C–105°C for 1 hour. After that, it was cooled in a desiccator for 20 minutes and weighed. The samples were then heated again in the oven for 1 hour, cooled back into the desiccator and weighed. This treatment is

repeated until a constant weight is reached.

#### *pH analysis*

pH analysis was carried out via the procedure of Said (2020), where a sample of 0.2 grams was weighed and dissolved in 20 mL of water. The samples were homogenized, and the degree of acidity was measured at room temperature using a pH meter.

#### *Analysis of the Ash Content*

The ash content was analyzed using the Association of Office Analytical Chemist (AOAC) method (1995). The results of the analysis of the water content revealed that the samples were heated in a furnace at a temperature of 660°C for  $\pm 3$  hours until they became ash.

#### *Viscosity Test*

The viscosity test was performed following the procedure of Sasmitaloka, et al. (2017). The gelatin solution with a concentration of 6.67% was heated on a hot plate at 80°C and then stirred until it was dissolved. A total of 20 g of the solution was measured for viscosity using a Rapid Visco Analyzer (Parten).

#### *Gel strength test*

The gel strength was measured following the procedure of Sántiz-Gómez et al. (2019). The gelatin solution was heated on a hot plate at 80°C, stirred until dissolution, and then put into a measuring cup. The samples were then stored at 10°C for 18 hours. Measurements were made using a Texture Analyzer XT-21 (Brookfield).

## RESULTS AND DISCUSSION

#### *Gelatin yield*

Table 1 shows that the concentration affects the yield value. This shows that the higher the concentration of phosphoric acid used, the greater the yield value obtained. The higher the concentration and duration of the immersion process are, the more hydrogen bonds and hydrophobic bonds are broken, which are the stabilizer bonds in the triple helix so that it becomes gelatin. The increase in yield was also influenced using acid, which resulted in the opening of the collagen structure to expand and open, along with the increase in concentration (Wiradana et al., 2020).

**Table 1.** Gelatin yield from Jerbung Shrimp Skin with three types of acid solvents

Sample	Concentration (%)	Solvent		
		Acetic acid	Phosphoric acid	Sulphate acid
Gelatin	1	1.21	8.80	0.91
	3	1.91	11.15	2.14
	6	2.06	12.90	2.65

***Gelatin pH***

The data in Table. 2 shows that the higher the concentration used, the more acidic the pH value. This occurred because the high concentration of phosphoric acid used causes many acids to stick to the shrimp shells during the soaking process. Therefore, the collagen that expands and is trapped in the fibril network is difficult to neutralize and is eventually hydrolyzed (Hidayat, et al., 2016: 75). The low pH value of gelatin is caused using strong acids during demineralization so that there are still remnants of acid bound to the extraction process, which can affect the acidity of the resulting gelatin. The pH values produced in this study were 5.74, 5.58 and 5.14. The results obtained are

quite good because the resulting pH value still meets the requirements of the British Standard, which is 4.5–6.5.

***Water content***

Data on the water content of the gelatin resulting from the synthesis of the three types of solvents can be found in Table 3. The moisture content is an indicator of the quality of food ingredients. The water content of a sample can affect microbial and chemical activity. The water content affects the quality of gelatin, such as the level of rancidity and resulting color (Juliasti et al, 2015: 8). The water content of gelatin obtained from three types of solvents met the requirements of SNI No. 06–3735 of 1995, with a maximum of 16%.

**Table 2.** Gelatin pH of Jerbung shrimp skin after treatment with three types of acidic solvents

Sample	Concentration (%)	Solvent		
		Acetic acid	Phosphoric acid	Sulphate acid
Gelatin	1	6.37	5.74	5.97
	3	5.96	5.58	5.89
	6	5.35	5.14	5.20

**Table 3.** Water content of gelatin from Jerbung shrimp shells in three types of acidic solvents

Sample	Concentration (%)	Solvent		
		Acetic acid	Phosphoric acid	Sulphate acid
Gelatin	1	10.65	3.30	12.35
	3	9.80	6.50	11.00
	6	8.60	5.70	9.20

**Table 4.** Gelatin ash content of Jerbung shrimp shells in three types of acidic solvents

Sample	Concentration (%)	Solvent		
		Acetic acid	Phosphoric acid	Sulphate acid
Gelatin	1	0.19	0.31	0.17
	3	0.16	0.33	0.14
	6	0.13	0.27	0.10

The ash content of shrimp shell gelatin (Table 4) is influenced by the large amount of minerals dissolved during the washing process. There is an internal component that is bound to collagen that has not been released during the washing and filtering process so that it is carried away during the ashing process (Febryana et al, 2018: 99). The results of the ash content analysis revealed that the 1995 SNI has a maximum ash content of 3.25%. The results of the measurements carried out met the standards.

### Viscosity

The results of the viscosity analysis are presented in Table 5. The data show that the higher the concentrations of acetic acid, phosphoric acid and sulfate acid used, the lower the viscosity of the gelatin. This is justified by the findings of Hong et al. (2017), who reported that the higher the concentration of acid used is, the more open the structure of the amino acid chain, which decrease the viscosity value. This is due to the use of acid, which influences

changes in the structure of collagen to spread or swell, causing the triple helix structure of collagen to change into a single chain structure. Changing the structure of the collagen chain causes a decrease in the molecular weight of gelatin. The results obtained in this study were only at a concentration of 6%, which met the quality standards required by SNI (1995), namely, the maximum viscosity limit for gelatin is 15–70 mps or 1.5–7 cP.

### Gel Strength

The results of this gel strength study (Table 6) revealed that the higher the acid concentration used was, the lower the gel strength value. This is due to the continued hydrolysis of collagen, which causes the covalent bonds that connect amino acids to one another to be broken, causing the molecular weight of collagen to decrease and shortening of the amino acid chains, resulting in a decreased gel strength (Santoso et al, 2015). The gel strength required by GMIA (2013) is between 50 and 300 blooms.

**Table 5.** Viscosity values of Gelatin from Jerbung shrimp shells in three types of acidic solvents

Sample	Concentration (%)	Solvent		
		Acetic acid	Phosphoric acid	Sulphate acid
Gelatin	1	6.74	13	6.40
	3	6.19	8	5.36
	6	5.81	7	5.05

**Table 6.** Strength of Gelatin from Jerbung shrimp skin with three types of acidic solvents

Sample	Concentration (%)	Solvent		
		Acetic acid (cP)	Phosphoric acid (cP)	Sulphate acid (cP)
Gelatin	1	116.04	155.85	109.06
	3	98.24	102.63	97.85
	6	92.63	85.37	94.15

**Table 7.** Characteristics of Gelatin Functional Groups from Jerbung shrimp shells with three types of acidic solvents

Sam ple	Absorption peak (cm <sup>-1</sup> )									Funct ional group
	Acetic acid (%)			Phosphate acid (%)			Sulphate acid (%)			
	1	3	6	1	3	6	1	3	6	
Gela tin	3419.79	3491.16	3491.16	3273,96	3281,40	3284,93	3403.36	3408.22	3406.29	O-H; N-H amida sekun der
	2960.73	2960.73	2960.73	2941,20	2942,23	2960,35	2960.73	2962.66	2929.87	C-H
	1651.07	1651.07	1651.07	1632,51	1628,50	1628,55	1654.92	1624.04	1624.06	C=O
	1585.45	1516.05	1516.05	1539,01	1539,41	1540,03	1517.98	1539.20	1541.12	N-H
	1240.23	1236.37	1234.44	1238,54	1239,77	1239,86	1126.43	1138.00	1010.70	C-O
	1076.28	1064.71	1064.71				1273.02			N-H bending

### FTIR analysis

FTIR spectral analysis aims to determine the functional groups of the resulting gelatin. Gelatin generally consists of hydroxyl groups (O-H), carbonyl groups (C=O) and amine groups (N-H) (Hassan et al., 2021). The results of the FTIR analysis of the gelatins from the Jerbung shrimp shells are shown in Table 7.

On the basis of the results in Table 7, the IR spectra of shrimp shell gelatin were obtained using three types of acidic solvents with concentrations of 1%, 3% and 6%. The results of the FTIR analysis of acetic acid gelatin revealed peaks at 3419.79 cm<sup>-1</sup>, 3491.16 cm<sup>-1</sup>, and 3491.16

cm<sup>-1</sup>; those of phosphoric acid gelatin were 3273.96 cm<sup>-1</sup>, 3281.40 cm<sup>-1</sup> and 3284.93 cm<sup>-1</sup>; and those of sulfuric acid gelatin were 3403.36, 3408.22, and 3406.29 cm<sup>-1</sup>. The absorption area is a functional group of amide A, which is characterized by O–H stretching and N–H stretching (amine groups) (Hassan et al., 2021). The absorption peak at a wavelength of 3200–3500 cm<sup>-1</sup> has a characteristic vibrational peak indicating the H stretching bond of O–H; in this absorption area, there is also an N–H stretching group (amine group), indicating the presence of gelatin.

The functional group absorption areas at wavenumbers were 2941.20 cm<sup>-1</sup>,



2942.23  $\text{cm}^{-1}$  and 2960.35  $\text{cm}^{-1}$ . The absorption area is the absorption area for the C-H functional group. The absorption peak of the C-H functional group is at a wavelength of 3000–2840  $\text{cm}^{-1}$ . The presence of a carboxyl group (C=O) in the wavenumber's range of 1600–1700  $\text{cm}^{-1}$ . This is in accordance with the FTIR analysis of each gelatin produced, which revealed that the absorption area at a wavelength of 1640–1820  $\text{cm}^{-1}$  indicates the presence of a functional group of carbonyl compounds, namely, C=O (amide), with a sharp absorption band and a weak intensity, indicating the presence of a functional group in the gelatin (Zhuo et al., 2017).

The functional group absorption area with an absorption area of 1560–1335  $\text{cm}^{-1}$  indicates the presence of functional group N-H bending from secondary amides and C-N stretching. The absorption area is the absorption area for the N-H bending functional group (C-N amine stretching). The typical group of gelatin, namely, amide III, has an absorption peak at 1240-670  $\text{cm}^{-1}$  (Hassan et al., 2021).

On the basis of the results of functional group analysis via the FTIR-ATR instrument, the compound produced from the skin of the Jerbung shrimp using sulfuric acid at concentrations of 1%, 3%

and 6% in this study is true gelatin because it has a functional group that makes up gelatin, namely, the O-H at wavenumbers 3600–3200  $\text{cm}^{-1}$ , the C-H group at wavenumbers 3000–2840  $\text{cm}^{-1}$ , the C=O group at wavenumbers 1800–1650  $\text{cm}^{-1}$  and the N-H group at wavelengths 1600–1460  $\text{cm}^{-1}$ . Although there is a slight difference in terms of the area or spectrum width indicated by each concentration, this difference can be influenced by the number of components present in each gelatin, such as differences in the amount of water content. On the basis of the results of all the tests, the use of different types of acid in gelatin synthesis is very possible. Gelatin can be produced through a heating method combined with a chemical method (Said 2020).

## CONCLUSION

Research has been conducted on gelatin using three types of acidic solvents, namely, acetic acid, phosphoric acid, and sulfuric acid. The pH, water content, ash content, and gel strength of the gelatin met the Indonesian National Standard (SNI) (1995). The characteristics of gelatin from Jerbung shrimp shells using FTIR-ATR revealed the presence of absorption areas from gelatin in the presence of functional groups, such as O-H groups at wavenumbers ranging from 3600–3200  $\text{cm}^{-1}$ , C-H groups at

wavenumbers ranging from 3000–2840  $\text{cm}^{-1}$ , C=O groups at wavenumbers ranging from 1800–1650  $\text{cm}^{-1}$  and N-H groups at wavelengths ranging from 1600–1460  $\text{cm}^{-1}$ . The characterization

results according to SNIs show that three types of acids can be used in gelatin hydrolysis. The synthesis of gelatin can be continued with the testing of other characteristics, such as solubility.

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