

Formulation and Evaluation of Formalin Washer Fluid Preparation from Garlic Peel Waste (*Allium sativum* L.)

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Abstract

Until 2022, there will still be many salted anchovies that contain formalin. This potential danger can be reduced by reducing the levels of formalin that may be contained in salted anchovies before consumption, one of which is by utilizing the saponin content in garlic peel waste. Garlic peel waste powder is proven to be able to reduce formalin levels in salted anchovies by up to 89.12%. However, the use of powder is less practical, and in a certain period, the powder can rot and emit an unpleasant odor. Therefore, an innovation is needed to transform the powder form into a formalin washer fluid. This study aims to determine the effect of garlic peel extract variation on several parameters evaluating the physical properties of formalin washing liquid. The extraction of garlic peel was done using the maceration method and a 70% ethanol solvent. Washing liquids were prepared using glycerin, PEG₄₀₀, EDTA, and distilled water with varying extract concentrations. Evaluation of physical properties included organoleptic, pH value, specific gravity, viscosity, clarity, and physical stability of formalin washer fluid. The stability test of the physical properties of the sample liquid was carried out using the cycling test method. The samples were kept in storage conditions with extreme temperatures for 3 cycles. In each cycle, the test preparation was stored for 24 hours at 4±2°C and 24 hours at 40±2°C. The formalin washer fluid produced was in the form of a liquid with a weak to pungent garlic aroma, yellow to dark brown in color, homogeneous, clear, and had a pH value of 4. The more extract is used in the formalin washer fluid, the darker the color and the more pungent the garlic aroma will be. Increasing the concentration of garlic peel extract does not affect the pH and clarity of the preparation. The higher the concentration of extract is, the higher the specific gravity and viscosity will be. The selected formula is the formula with a 1% concentration of garlic peel extract.

Keywords: *Allium sativum*; formalin; formulation; peel; wash liquid

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INTRODUCTION

As a city of education and culture, Yogyakarta is also known as a paradise for culinary lovers for both domestic and foreign tourists. One of Jogja's culinary

specialities is *angkringan* cat rice. It encourages the growth of *angkringan* outlets in Yogyakarta to reach two thousand units every year.¹ An *angkringan* seller can use 1.5kg of salted anchovies to sell cat rice.² Although this culinary is

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much sought after, many studies showed that salted anchovies contain formaldehyde as raw materials for making cat rice.³ It has been the case for most of the last 10 years, both in Yogyakarta and other cities in Indonesia, such as Bandung, Surakarta, Palembang, and Medan.⁴⁻⁹

Burhan's research¹⁰ demonstrated that up to 84% of salted anchovies circulating in traditional markets in Yogyakarta still contained formalin, with the highest concentration of 12.65%. Similar conditions were also found in Yogyakarta City in 2022, where 100% of salted anchovy rice samples were positive for formalin, with the highest concentration of 11.75%.¹¹ It exceeds the IPCS threshold for formaldehyde of 1 ppm.¹² When people consume formaldehyde, it can cause health problems such as sore throat, nausea, vomiting, diarrhea, bleeding, headache, hypotension, convulsions, liver, heart, brain, spleen, pancreas, central nervous system, and kidney damage, and in the long term, it can cause cancer.¹³

Food safety issues such as formaldehyde abuse in anchovies are one of the priorities of the 2017-2045 national research plan, especially in the fields of health and medicine. Indonesia's food safety status is still low, ranking 76 out of 105 countries, due to the abundance of hazardous chemicals such as formaldehyde in food.¹⁴ In line with that, Poltekkes Bhakti Setya Indonesia also supports the government's efforts to improve food safety through food pharmacy and public health.¹⁵ Based on this, it is necessary to improve food safety against the potential hazards of formalin in anchovies before humans consume the product.

The problem of salted fish with formalin can be solved by using natural ingredients that contain saponins. Among them, the

use of cassava leaf extract aims to reduce white shrimp formalin levels, aloe vera to reduce formalin in milkfish and tofu, miana leaves to reduce tofu formalin, and pandan leaves to reduce formalin levels in black grass jelly and salted anchovy rice.^{8,16-20} In addition to these ingredients, kitchen waste such as garlic peel has also been shown to be able to reduce formaldehyde levels in salted anchovy rice up to 89.12%.¹¹ It is because garlic peel also contains the active compound saponin.²¹ Indonesia has been identified as a country with a high opportunity for garlic peel waste utilization. According to the Indonesian Central Bureau of Statistics, in 2021, Indonesia can produce forty-five thousand tonnes of garlic, while the D.I. Yogyakarta and Central Java region can produce twenty-five thousand tonnes of garlic.²²

Saponin compounds can bind formalin particles to dissolve in water. Saponin is a soap substance (surfactant) with two polar and non-polar groups that can form emulsions. The process of emulsion formation from emulsifying compounds (surfactants or surfactants) is called saponification.¹⁶ The presence of emulators can stabilize the froth due to a decrease in water surface tension. Formalin bound to the product can be released by the action of polar (hydrophilic) and non-polar (hydrophobic) groups on the surfactant of the saponin compound. The substance adsorbs in the area between the phases and binds to the formaldehyde particles in such a way that emulsion stability is achieved by the polar groups.²³

However, Burhan's research has proven that garlic peel waste powder is able to reduce formaldehyde levels in salted anchovy rice by up to 89.12%.²³ However, it still has several disadvantages, including

impracticality in use. In addition, long-term storage of powder can cause a musty odor. A product must not only have quality and benefits but also be acceptable and desirable to its users. Another great potential is the abundance of garlic peel waste in Indonesia.²² If the weight of garlic peel waste is $\frac{1}{20}$ of the total weight, then in one year, Indonesia can produce 2,500 tonnes of garlic peel waste. This abundance of waste can be used to produce extracts that are then used as raw materials for the manufacture of formalin wash liquid.

This study aims to determine the effect of garlic peel extract variation on several physical properties evaluation parameters, including organoleptic, pH value, specific gravity, viscosity, clarity, and physical stability. The results of the evaluation of the physical properties of this washing liquid can later determine the selected formula so that it will become a reference formula for other natural ingredients that also have the potential to reduce formalin in food ingredients.

METHOD

Materials

Tools used to make formalin washer fluid included a basin, blender, measuring cup, analytical balance, sieve no.mesh 20 and 40, glass Bekker, macerator, wooden stirrer, porcelain cup, mortar, Stemper,

and water bath. Materials used to make formalin washer fluid included garlic peel, 70% ethanol, distilled water, glycerin, EDTA, and PEG 400.

Preparation of garlic peel extract

Garlic peel was dry sorted by selecting clean, non-moldy simplisia, marked with a pure white color. Powder preparation was carried out using a blender, then sieved with a $\frac{20}{40}$ mesh number sieve. The powder obtained was extracted with 70% ethanol (1:10) using the maceration method followed by remaceration. The liquid extract obtained was evaporated in a water bath until it became a thick extract.

Formulation of formalin washer fluid from garlic peel extract

Preparation of formalin washer fluid was done by mixing ethanol extract of garlic peel with glycerin until homogeneous. Polyethylene glycol (PEG) 400 was added to the mixture (phase A). In a different container, EDTA was dissolved in a portion of distilled water (phase B). Phase A was added little by little to phase B and stirred until homogeneous, then the remaining distilled water was added. The evaluation was carried out in the form of organoleptic, homogeneity, and pH value. The formalin washer fluid formula is shown in Table 1.

Table 1. The formula of formalin washer fluid from garlic peel extract

| Composition | Concentration (% ^b / _b) | | | | |
|---------------------|--|--------|--------|--------|--------|
| | Base | F1 | F2 | F3 | F4 |
| Garlic peel extract | 0 | 0.1 | 0.5 | 1 | 5 |
| Glycerin | 5 | 5 | 5 | 5 | 5 |
| PEG 400 | 10 | 10 | 10 | 10 | 10 |
| EDTA | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 |
| Distilled water | Ad 100 | Ad 100 | Ad 100 | Ad 100 | Ad 100 |

Evaluation of formalin washer fluid from garlic peel extract

Organoleptic

The organoleptic examination was performed visually by observing the shape, color, taste, and odor of the preparation.

pH value

Universal indicators will give a certain color if dripped or dipped into an acidic or basic solution. The formed color will be matched with the color of the known pH value.²⁴ The recommended pH value for food-grade liquids ranges from 4-7.

Specific gravity

The first stage of determining specific gravity is to determine the volume of the pycnometer at the experimental temperature. Next, a clean and dry pycnometer was weighed carefully. The pycnometer was filled with sample liquid to the brim and then immersed in ice water so that the temperature was approximately 2°C below the experimental temperature (25°C). The pycnometer was closed and removed from the ice water, the capillary tube was left open, and the temperature was allowed to rise to the experimental temperature (25°C); then, the capillary tube of the pycnometer was closed. The pycnometer containing the sample liquid was allowed to stand until it reached room temperature, and then the dew water attached was cleaned and weighed carefully.

Viscosity

Determination of sample viscosity was carried out with an Ostwald viscometer. A 10mL sample was introduced into the Ostwald viscometer using a suction pipette until it passed through two boundaries. The time taken by the liquid to

pass through two predetermined points on a vertical capillary tube was measured as flow time. The viscosity of the sample is determined by comparing the flow time of the solvent and sample solution in centipoise (cps).

Clarity

The clarity test examination was carried out visually using a clean container and then examined from the outside under good lighting, using a black background obstructed against reflection in the eye. The preparation must be completely free of small visible particles.

Stability test by cycling test

The stability test of the physical properties of the sample liquid was carried out using the cycling test method. The samples were stored in storage conditions with extreme temperatures for 3 cycles. In each cycle, the test preparation was stored for 24 hours at 4°C and 24 hours at 40°C.²⁵ Physical properties test observations were made before and after storage in the form of organoleptic, pH value, specific gravity, viscosity, and clarity.

RESULTS AND DISCUSSION

The preparation of garlic peel extract was done by sieving with a 20/40 mesh number sieve, indicating that the powder used was a powder that passed the 20mesh sieve but did not pass the 40mesh sieve. This method aims to produce a powder that was similar in size, which was not too coarse, and not too fine. Powder that was too coarse would cause a decrease in the effectiveness of the active substance extraction process into the solvent.²⁶

Saponins from garlic peel were extracted using 70% ethanol as saponins could dissolve in fairly polar solvents such as 70% ethanol, which contained 30% water.

Maceration is an applicable, simple extraction method commonly used in large and small pharmaceutical industries. The extraction yield obtained was 3.8%. The resulting extract was thick, yellowish brown in color, with a distinctive aroma of garlic (Figure 1).



Figure 1. Ethanol extract of garlic peel

Formulation of formalin washer fluid from garlic peel extract

The basic ingredients in formalin washer fluid preparations are thickeners, cosolvents, and solvents. Additional ingredients include preservatives and complexing agents. The thickener used is glycerin due to its ability to increase viscosity, stabilize, be non-toxic, non-irritating, and be safe when in direct contact with food or drink.²⁷ The cosolvent used is polyethylene glycol (PEG) 400 as it can significantly increase the solubility of active substances in water, has a low molecular weight, is in the form of a clear, colorless, viscous liquid, and has low toxicity.²⁸ The solvent used is water. EDTA (Ethylenediamine tetra-acetic acid) material is used as a stable complexing agent as EDTA is a polydentate ligand that has six pairs of free electrons so that it can grip the central atom very strongly. It will form stable complexes with metals that may be present in the production equipment and storage containers, except for sodium and calcium. A concentration

of 0.01-0.1% EDTA can be used as a preservative for liquid preparations.²⁹

In the preparation of formalin wash liquid, it needs to be divided into two phases based on the solubility of each ingredient. Active ingredients such as garlic peel extract require cosolvents to increase their solubility in water, so they need to be mixed first with cosolvents such as PEG 400 and glycerin. Ingredients such as EDTA are easily soluble in water so that they can be dissolved directly in distilled water. This method can overcome the problem of incompatibility of the ingredients used. Thus, when the two phases are mixed, it will produce a homogeneous liquid preparation.

The formulation of the washing liquid was carried out by varying the concentration of garlic peel extract at a concentration of 0.1% to 5%. This level was chosen based on the research of Burhan, Irianto, Wijaya, Pradah, et al.²³ showing that garlic peel powder was able to reduce formalin in salted anchovies by 89.12%. Liquid preparations were chosen as they are easy to apply to food ingredients. The diffusion process of active substances such as saponins to food ingredients is easier and faster, to ease the formalin reduction in food ingredients.

Evaluation of formalin washing fluid from garlic peel extract

The formalin washing liquid produced is a liquid with a weak to pungent garlic aroma, yellow to brown in colour, homogeneous, clear, and has a pH value of 4 (Tables 2 and 3). This pH value is in accordance with the recommended pH value requirements for consumption, such as herbal drinks, which range 4-7, included in the food-grade category.³⁰

Table 2. Organoleptic test results of formalin washer fluid formula of garlic peel extract

| Parameters | Base | F ₁ | F ₂ | F ₃ | F ₄ |
|-------------|-------------------|----------------------------------|--|---|-----------------------------|
| Color | Clear | Yellow | Brownish-yellow | Brown | Deep brown |
| Odor | Typical weak odor | Typical weak odor of garlic peel | Medium distinctive odor of garlic peel | Strong characteristic odor of garlic peel | Pungent odor of garlic peel |
| Dosage form | Liquid | Liquid | Liquid | Liquid | Liquid |
| pH value | 4 | 4 | 4 | 4 | 4 |

Figure



Based on the organoleptic test of formalin washer fluid, the more extract is used, the more brown the color of the liquid and the more pungent the smell of garlic will be. It is because the compounds responsible for the color, taste, and smell of garlic peel are increasingly extracted. These include sulfur, thiosulfate, alliin, or S-allyl-cysteine sulfoxide (ACSO), which are responsible for flavor precursors as well as properties of garlic peel such as antibacterial, antifungal, antioxidant, and antidiabetic.³¹⁻³³ In addition, garlic peel also contains ajoene and allicin, which are responsible for the distinctive and strong aroma. Ajoene is an unsaturated sulfide formed from three allicin molecules bonded together and can prevent blood clots.³¹

Based on the results of the specific gravity and viscosity tests of formalin washer fluid, the higher the concentration of garlic peel extract is used, the higher the specific gravity and viscosity will be (Figure 2). Specific gravity is related to the weight fraction of the components contained in a liquid. The greater the weight fraction of a liquid is, the greater the specific gravity value will be.³⁴ The higher the concentration of garlic peel extract in the washing liquid formula is, the more compound components are quantitatively contained in it so that the weight fraction gets bigger and the higher in the specific gravity. These results are in line with the research of Suhendy et al.³⁴ which showed that the higher the specific gravity value is, the higher the total flavonoid content will be.

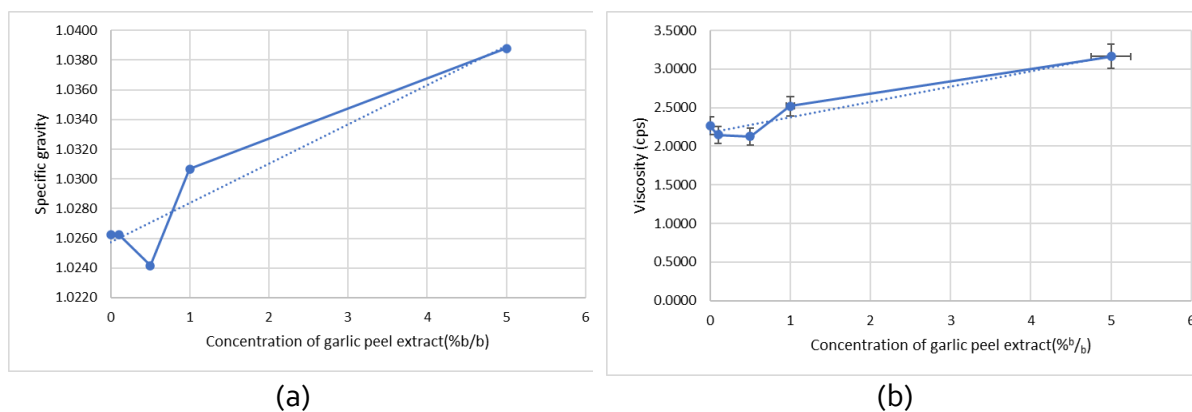


Figure 2. Relationship between concentration of garlic peel extract on specific gravity (a) and viscosity (b).

The results of the formalin washer fluid stability test were carried out using the cycling test method in order to evaluate the characteristics of the preparation under extreme storage conditions (temperature $4\pm 2^{\circ}\text{C}$ and $40\pm 2^{\circ}\text{C}$) so as to show the stability of the preparation during the distribution process.³⁵ Odor, color, shape, and pH value did not change during extreme storage conditions. These results indicate that the compound content in garlic peel is stable at $4\pm 2^{\circ}\text{C}$ and $40\pm 2^{\circ}\text{C}$.

The higher concentration of garlic skin extract in the washing liquid did not affect the clarity of the preparation. However, extreme storage conditions affect the clarity and stability of the preparation. Base formulas with extract concentrations of 0.1% and 0.5% showed white particles such as microbes floating on the

preparation. It shows that the concentration of EDTA used is not enough to resist microbial growth of the washing liquid preparation. The higher the concentration of the extract is, the more stable the clarity of the formalin washer fluid will be (clear). It indicated that the formalin washer fluid with 1% and 5% concentrations of garlic skin extract is able to prevent microbial growth of the preparation at extreme conditions, temperatures $4\pm 2^{\circ}\text{C}$ and $40\pm 2^{\circ}\text{C}$. This ability can occur as garlic skin extract also has antibacterial activity (11,35). The active ingredients responsible for antibacterial activity include saponins and allicin. Saponin is able to induce bacterial cell leakage, while allicin is able to inhibit bacterial RNA synthesis and interact with enzymes containing thiol groups.³⁶

Table 3. Clarity stability test results of formalin washer fluid formula of garlic peel extract

| Treatment | Clarity | |
|-----------|----------------|-------------------------|
| | Before storage | After storage |
| Base | Clear | White particles present |
| F1 | Clear | White particles present |
| F2 | Clear | White particles present |
| F3 | Clear | Clear |
| F4 | Clear | Clear |

After storage, the weight fractions of the formalin-based garlic skin extract washes tended to be stable (Table 4). Changes in extreme storage temperature conditions did not affect the weight fraction of the components contained in the resulting preparation. However, the viscosity was

not stable at these extreme storage temperatures. This condition indicates that the ability of glycerin, which is responsible for the viscosity of the preparation, is affected by storage temperature.

Table 4. Stability of test results of specific density and viscosity of formalin washer fluid formula of garlic peel extract

| Treatment | Specific Gravity | | Viscosity (cps) | |
|-----------|------------------|---------------|-----------------|---------------|
| | Before storage | After storage | Before storage | After storage |
| Base | 1.0262±0.0008 | 1.0267±0.0012 | 2.2649±0.0522 | 2.1861±0.1099 |
| F1 | 1.0263±0.0012 | 1.0278±0.0008 | 2.1484±0.1110 | 2.2790±0.0217 |
| F2 | 1.0242±0.0024 | 1.0275±0.0006 | 2.1268±0.0624 | 2.2039±0.1316 |
| F3 | 1.0307±0.0005 | 1.0319±0.0001 | 2.5199±0.0759 | 2.2693±0.0721 |
| F4 | 1.0388±0.0016 | 1.0395±0.0031 | 3.1633±0.0316 | 3.3215±0.0232 |

Based on the evaluation results, the selected formula is F₃, which is a formula with a concentration of 1% garlic skin extract. This selection is based on all evaluation parameters, especially having stable clarity, not overgrown with microbes, and preparation color that is not too concentrated and not too thick.

CONCLUSION

The more extract is used in the formalin washer fluid, the darker the color and the more pungent the aroma of garlic will be. Increasing the concentration of garlic skin extract did not affect the pH and clarity of the preparation. The higher the concentration of extract is, the higher the density and viscosity will be. The selected formula was the formula with a 1% concentration of garlic skin extract.

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