Chitosan membrane technology as adsorbent media: management of Tallo River Basin Makassar City, South Sulawesi, Indonesia

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Abstract. This study aims to determine the surface area and adsorption power of the chitosan membrane against Pb (II) metal ions in the Tallo river basin and determine the optimization of Pb (II) ion absorption by chitosan membrane adsorbents and the adsorption time of the chitosan membrane against Pb (II) metal ion in Tallo river basin. The method used in this study consisted of chitosan synthesis from crab shell waste, chitosan membrane design with various membrane variations comprised of a concentration ratio of chitosan and cellulose acetate respectively 35%, 65%, 45%: 55%, 50%: 50% and 65%: 35% with variations in adsorption time of 10; 25; 45; 60 minutes, then followed by the adsorption mechanism of the chitosan membrane against Pb (II) metal ions. Determination of the surface area was carried out using the Brunner Emmer Teller (BET) analysis. In contrast, the optimization of Pb (II) ion absorption and adsorption power was carried out using UV Vis spectrophotometry. The results showed that the surface area of the chitosan membrane reached 765 m²/g, a pore volume of 1,267 cc/g, and a pore diameter of 4.7 nm, the adsorption capacity of the chitosan membrane against Pb (II) metal ions was 76.34%. In comparison, the adsorption capacity of the chitosan membrane against ions Pb (II) metal was 68.5 mg/g for 100 minutes. This study recommends the use of a chitosan membrane to reduce the levels of Pb (II) metal ions and improve the quality of Tallo river water for consumption.

1. Introduction
The phenomenon of environmental pollution, especially river water quality, is related to global climate change. Global climate change affects watersheds caused by rainfall, rising temperatures, and waves of human activity and pollution [1,2]. The watershed is an area that is very vulnerable to pollution caused by various factors. Development of urban areas, agricultural activities, mining, and industrial activities have contributed significantly to the decline in water quality in the watershed area [3].
Water pollution in the watershed can be in heavy metal ions from various human activities that can cause toxicity to humans [4]. Some heavy metals such as cadmium, mercury, lead-silver, and arsenic impact human health, namely respiratory disorders, nervous system damage, defects in infants, carcinogenicity, and impaired immune function [5]. Furthermore, exposure to lead (Pb) has an impact on kidney damage [6].

Various methods to eliminate river water pollution have been widely developed, such as membrane bioreactor electrochemical systems, denitrifying biofilters, and disinfection technologies [7]. The bioreactor membrane has a more complex design consisting of a two-part reactor system [8]. Disinfection techniques to reduce pollution levels in river water have also been developed using an integrated system method [9]. In addition, the denitrification method produces a denitrification coefficient which increases with time [10].

The use of chitosan as an adsorbent media in the water treatment process is an alternative that can be developed, especially in watersheds. Chitosan can be used as an adsorbent medium because it is a deacetylation process of chitin that comes from waste shrimp shells, crab shells, and insects [11,12]. Chitosan has antibacterial and antifungal properties and has been widely studied as a potential natural antimicrobial agent in the pharmaceutical, cosmetic, agricultural, and food industries. Chitosan can be combined with other polymers to increase its effectiveness [13,14]. An amino group's presence in the chitosan structure distinguishes chitosan from chitin and gives this polymer many unique properties [15]. The binding of chitosan with triploypostate material forms a durable, biodegradable plastic [16].

The coagulation process to remove impurities in river water can be done by adding chitosan compounds [17]. Chitosan, which reacts with polylactic acid and chloroform in PEG, gives Escherichia coli and staphylococcus antibacterial properties more flexible membrane characteristics [18]. The wastewater coagulation process using chitosan and poly aluminum chloride produces high purity wastewater [19]. The adsorption method using a mixture of chitosan and acetic acid and tetrachloride provides a relatively more negligible absorbance so that it can be used to adsorb toxic compounds [20]. The adsorption method of chitosan by adding a salt such as aluminum sulfate has reduced the level of pollution in wastewater which is influenced by an increase in the degree of deacetylase in chitosan [21].

The focus of this study is aimed at answering the questions: (i) how the surface area and adsorption power of chitosan membrane to Pb (II) metal ions in Tallo watershed (ii) how the optimization of Pb (II) ion absorption by chitosan membrane adsorbent and chitosan membrane adsorption to Pb (II) metal ions in Tallo watershed. It was conducted by testing Pb (II) metal ion samples at 3 (three) sampling locations to test the water quality of the Tallo river in Makassar city.

2. Methodology

2.1 Profile of study area

Geographically, Tallo River Basin is located between coordinates 5°6'-5°16' South Latitude and 119°3'-119°46' East Longitude. It passes three administrative areas of the city and regency, i.e., Makassar, Gowa, and Maros, with 407 kilometers. The existence of the Tallo River from upstream to downstream is 72.00 kilometers, with a total watershed area reaching 339.90 kilometers. Tides influence the estuary of this river. Tides that occur are mixed wave prevailing semi-diurnal, with an asymmetrical tidal curve shape, i.e., tide time of 8-9 hours, tidal time of 14-16 hours, and slack time of 2-3 hours. Tidal conditions affect the current velocity of the river heading to the sea. The tide flows upstream of the river, and the tidal flows to the estuary. The maximum current velocity reaches 0.254 m/s. The bathymetry conditions of Tallo River based on the lowest tide during the survey were at -70.40 cm from the water surface [22].

The selection of sampling locations at 3 (three) sites was based on considerations: (i) The upper part of Tallo River is influenced by the presence of the Electric Steam Power Plant (PLTU) industry, which the waste discharges directly into the Tallo river body, (ii) the central part of Tallo river is
contacted directly with the residential settlements, hospitals and SMEs, and (iii) the lower part of Tallo river is contacted directly with the Makassar Industrial Estate (KIMA). The coordinate for sampling locations is shown in Table 1 and the condition of the Tallo watershed in figure 2.

**Table 1. Sampling sites of Tallo River water quality.**

| Water Sampling                      | Coordinate Point                                      | Potential of Water Quality                       |
|------------------------------------|-------------------------------------------------------|--------------------------------------------------|
| The upper part of Tallo River      | 119°37'71.347"E 5°08'15.138"S                         | Industrial Area, PLTU                            |
| (PLTU Tallo area)                  |                                                       |                                                  |
| The central part of Tallo River    | 119°43'07.870"E 5°12'03.052"S                         | Settlement, Hospital, and SME                    |
| (Pampang)                          |                                                       |                                                  |
| The lower part of Tallo River      | 119°43'07.878"E 5°12'03.052"S                         | Makassar Industrial Estate (KIMA)                |
| (TP1 Bridge)                       |                                                       |                                                  |

**Figure 1.** Map of the sample distribution of Tallo River water quality.

**Figure 2.** A, B. Example, Sampling sites of Tallo River.

Figure 2 shows the condition and bed elevation of the river. The length of the river reaches 4.30 km. The average width of the river reaches 48.70 m with a minimum width of 35.00 m and a maximum of 60.00 m. The height of the Tallo river water level is highly dependent on the intensity of rainfall and tides that occur. In the rainy season, the maximum water discharge reaches 79,685 m³/s. The water level reaches 3.18 m from the riverbed. In the dry season, the minimum flow is 21,141 m³/s. The water level
is around 1.66 m from the river bed. The contour of the Tallo River has no islands, so there are no obstacles during river water drainage. Sampling was conducted with a purposive sampling technique by specific considerations based on consideration of the dominant polluted river points at 3 (three) location points, namely (i) point 1 in PLTU Bridge in the upper part of Tallo River, (ii) point 2 in Pampang in the central part of Tallo River, and (iii) point 3 in Toll Bridge in the Lower part of Tallo River.

2.2 Data collection
In this study, data collection was divided into 2 (two) categories, namely (i) primary data obtained through preliminary surveys in the field, including the general condition of the study site, potential sources of pollution (river/channel confluence), river profiles, and observations/measurements in the laboratory, and (ii) secondary data based on the literature used as a basis and reference.

2.3. Experiment

2.3.1. Synthesis of chitosan from crab shells. Deproteinase stage: 50 gram of crab powder sample was put into a 500 ml three-necked flask, then 4% NaOH was added with a ratio of sample concentration to NaOH of 1: 10 w/v. The mixture is then heated and stirred at 80°C for an hour under reflux conditions. Then, the mixture is filtered using filter paper, and the residue has been washed using distilled water until neutral. After the neutral conditions are reached, the residue obtained is dried at 60°C for 48 hours.

Demineralization stage: 10 grams of deproteinized residue was put into a 600 ml beaker, then added 1 M HCl with a ratio of sample concentration to HCl of 1:15 w/v. The mixture is then stirred at room temperature for 3 hours. Then, the mixture is filtered using filter paper, and the residue in the form of chitin has been washed using distilled water until neutral. After the neutral conditions are reached, the chitin obtained is then dried at 60°C for 48 hours.

Decolorization stage. Chitin produced through the demineralization process is added with H2O2 to whiten the chitin to produce a quality product. The conversion of chitin to chitosan is done by enzymatically using the enzyme chitin deacetylase. The chitosan produced is then used to make chitosan membranes.

2.3.2. Making chitosan membrane. Chitosan membrane synthesis uses the phase inversion method with solvent evaporation at a drying temperature of 80°C. Chitosan membrane is made with a variable ratio of chitosan: cellulose acetate composition of 35:65, 45:55, 55:45, and 65:35 (v/v) with stirring variations at 150 rpm and 200 rpm. Chitosan membrane with a variation ratio of 35:65 (v/v) and a speed of 150 rpm was made by adding 0.25 gram cellulose acetate to a mixture of 35 ml chitosan 5% (w/v) and 65 ml cellulose acetate 5% (w/v) in stirring 150 rpm until homogeneous. The solution is then poured on a petri dish and dried at 80°C for 24 hours to form a membrane layer. The removal of the membrane was done by soaking 1% (w/v) NaOH for two hours. The membrane is then washed using distilled water before used in an adsorption application.

2.3.3. Adsorption procedure of chitosan membrane. 10 mg of adsorbent are immersed in a 15 mL solution from the Tallo river water sample and then centrifuged with a shocking rate of 100 rpm at 25°C. The pH value is controlled by 0.1 mol/L H2SO4 or 0.1 mol/L NaOH solution. The adsorption process lasts for 24 hours with a Pb (II) concentration of 200 mg/L at room temperature.

2.4 Analysis method

2.4.1 Testing of adsorption membrane. Determination of surface area, adsorption power, adsorption capacity, and absorption time of chitosan membrane to absorb Pb (II) metal ions in Tallo river water. Determination of surface area of pore volume and size is characterized by N2 adsorption and desorption using Brunauer-Emmett-Teller (BET, Autosorb-iQ2, Quantachrome Instruments, Shanghai, China) analyzer. The composition on the CS membrane surface was tested by XPS (ESCA Lab220i-XL). The membrane to be stretched was circled-cut with a diameter of ± 7 cm.
Then, the membrane is placed at the bottom of tester equipment previously coated with filter paper. One hundred milliliters of Pb (II) solution is put into the tool, tightly closed, and flowed in pressure 1-5 kg/cm$^2$. The resulting permeate volume is recorded every 5 minutes for 30 minutes. Pb (II) adsorption isotherms are calculated through initial concentrations in the range of 50-500 mg/L. The amount of adsorption $q$ (mg/g), Pb (II) for each adsorbent, chitosan is determined based on the formula:

$$q = \frac{c_0 - c_e}{m} \times V \quad (1)$$

$C_0$ and $C_e$ (mg/L) are the initial concentration and balance concentration of Pb (II) in solution, respectively. $V$ (L) is the explanation volume, and $m$ (g) is the initial membrane weight.

3 Results and discussion

3.1. Determination of surface area and adsorption power of chitosan membrane

Chitosan is a natural biopolymer derived from crustaceans, shrimp, bacteria, and fungi wastes. The use of chitosan membranes in the process of adsorption of Pb (II) metal ions is very beneficial by supporting enormous chitosan membrane adsorption power and the presence of amine groups on chitosan membranes that can bind covalently to metal ions in water samples. Determination of surface area and adsorption power aims to improve the quality of chitosan membranes to adsorb Pb (II) metal ions in Tallo river water samples. The more surface area of the membrane, the greater the ability to bind Pb (II) ions. While the adsorption power aims to determine how much the ability of chitosan membranes to absorb Pb (II) metal ions in Tallo river water samples.

3.1.1. Values of surface area and adsorption power. Determination of surface area using the BET method to determine the surface area, pore-volume, and pore diameter of the chitosan membrane. The BET test results for modifying the chitosan cellulose acetate membrane showed that the larger surface area of the chitosan membrane reached 765 m$^2$/g, a pore volume of 1.267 cc/g a pore diameter of 4.7 nm. This indicates that the modified chitosan membrane has membrane permeability properties and more extraordinary membrane absorption ability. The surface area and pore diameter on the modified chitosan membrane of polyethylene glycol is higher than pure chitosan membranes so that the membrane permeability is more excellent [23]. A mixture of chitosan-gelatin and ferulic acid with acidic solvents giving a larger pore dimension to form a larger membrane thickness. [24]. The large surface area of the activated carbon fiber of the lignin compound is proportional to the pore diameter and pore capacity of the membrane [25].

| Membrane Type | Surface area m$^2$/g | Pore Diameter Nm | Pore Volume cc/g |
|---------------|----------------------|-----------------|-----------------|
| A             | 237                  | 10.7            | 0.879           |
| B             | 451                  | 8.2             | 1.015           |
| C             | 674                  | 6.5             | 1.125           |
| D             | 765                  | 4.7             | 1.267           |
Chitosan membrane adsorption power to Pb (II) metal ions was calculated by evaluating the concentration before and after the adsorption process (figure 4). Chitosan membrane used to adsorb Pb (II) metal is a sample that has a surface area of 765 m$^2$/g, pore volume of 1,267 cc/g, and pore diameter of 4.7 nm. Pb (II) every 100 ml was adsorbed with a 0.1 gram chitosan membrane sample with an initial concentration of 100 ppm Pb (II). The research results obtained that the maximum adsorption power of chitosan membrane to Pb (II) ions was 83.26% pada Waktu adsorption 25 minutes. The increase in adsorption power increases with increasing adsorption time subsequently decreases because the empty site on the membrane has become saturated. The adsorption power in the activated carbon adsorption process increases with increasing adsorption time and decreases with increasing adsorbate concentration [26].

Chitosan Membrane Adsorption Power

3.2. Optimization of Pb (ii) adsorption by chitosan membrane adsorbent

Concentration optimization aims to determine the optimum Pb (II) ion concentration adsorbed by chitosan membrane adsorbents. The results showed that the greater of chitosan concentration, the greater of adsorbed metal concentration, and the greater chitosan absorption. Figure 8 shows the increase in the adsorbed Pb (II) metal ion concentration at the optimum absorption of 55% with the number of adsorbed Pb (II) metal adsorbed at 68.5 mg/g adsorption capacity at 25.83 mg/g for 25 minutes. The increasing initial concentration of chitosan membrane increases the absorption of Pb (II) metal ions. This happens because in the initial conditions, the absorption ability of the chitosan membrane is still low, so it has not been able to bind metal ions Pb (II) effectively. The optimum absorption occurs at a concentration of 55%. In this situation, the surface adsorption capacity of chitosan has been saturated, and equilibrium has been reached between the concentrations of metal ions in chitosan with its environment so that absorption at concentrations above 55% becomes constant or almost the same. The adsorption occurs because the surface of the adsorbent experiences a force imbalance; thus, the surface of the adsorbent easily attracts other substances so that the force balance is achieved. Chitosan adsorption power is caused because chitosan has large pores, and adsorption
occurs due to the potential energy difference between the surface of chitosan and the adsorbed substance. Chitosan used as an adsorbent can adsorb Pb (II) metal. This is because chitosan is a cellulose compound in which the resulting carbon atoms are bound to form a hexagon structure with carbon atoms located at each corner. The adsorption power of chitosan mixed with aniline with the hexagon structure on the recovery of Cu (II) ions gives a more excellent value of adsorption capacity and adsorption kinetics and adsorption capacity [27].

Figure 5. Optimization of adsorption of Pb (II) ion concentrations by chitosan membranes.

Structuring between layers and hexagonal rings owned resulted in the availability of spaces in the structure of chitosan that causes adsorbents to enter the porous chitosan structure. The mechanism in adsorption is adsorbate molecule diffuses through the boundary layer to the outer surface of the adsorbent, some of which is adsorbed on the outer shell, and most of it diffuses further into the adsorbent pores. If the adsorption capacity is still significant, most will be adsorbed on the surface. To determine the maximum adsorption capacity that can be adsorbed by chitosan to Pb (II) metal ions. Determination of adsorption capacity using the Langmuir isotherm model was studied on the adsorption isotherm curve created by plotting the concentration of metal ions in equilibrium versus the number of metal ions adsorbed. Pb (II) adsorption isotherm curves on chitosan adsorbents show the maximum adsorption capacity price of chitosan can absorb Pb (II) metal ions according to Langmuir isotherm of 68.5 mg/g with a regression coefficient of 0.986 and equilibrium constant value $b = 0.0005$ (figure 4). The adsorption kinetics of Pb$^{2+}$ ions follows the Freundlich isotherm equation. The adsorption capacity increases with increasing adsorption time while the adsorption capacity decreases with increasing adsorbate [28].

3.3. Mechanisms of Pb (II) ion adsorption on Tallo River Basin (chitosan membrane adsorbent)

Chitosan membrane adsorbent with a contact time of 100 minutes reduced the metal ion content of Pb (II) in the Tallo river water sample from a concentration of 43 mg/l to 0.187 mg/l. This decrease is substantial, reaching 98.5%, and is a relatively similar decline. The highest reduction in Pb (II) content, namely the lead (Pb) compound content, the highest reaches 97.5% at chitosan: cellulose acetate concentration 55%: 45% with a contact time of 100 minutes. This shows that chitosan adsorbent can adsorb Pb (II) ions maximally due to the bond between the amine groups in chitosan with positive ions in Pb (II) ions. The absorption ability of Hg (II) and Pb (II) metal ions from the biomaterial occurs in the adsorption-desorption mechanism [29]. The decreasing of Pb (II) metal ions concentration will improve the water quality of Tallo river and making it suitable for consumption to meet the drinking water needs of Makassar residents.
3.4. Solution of water resource management in Tallo River

Water resource management in Tallo River is related to efforts to control the potential of pollutants on water quality. Various pollutant sources originating from industrial activities such as PLTU, Makassar Industrial Zone (KIMA), residential settlements, and waste disposal from various Small and Medium Enterprises (SMEs) contribute positively to the decline in river water quality, in the sense that water conditions contain hazardous metals (Pb (II) metal ion). This fact causes the water quality of the Tallo River to be not suitable for consumption. Therefore it is necessary to take precautions and control the quality of water for various activities around it.

Therefore, prevention and quality control of water quality is needed for various activities around it. Precaution that can be taken includes (i) requiring wastewater management for various city activities by preparing water treatment before being channeled to water bodies, (ii) controlling the utilization of river banks with a distance of 40-50 meters by issuing urban activities that are located in river benefit areas, (iii) restrictions on city activities through zoning arrangements for river benefit areas followed by conservation of land and water resources, (iv) building security structures on the left and right sides of the river to prevent river erosion and sedimentation, and (v) socialization to the community not to use the river body like a garbage disposal media. These five things need government policy support, private involvement, and community participation. The interconnection of anthropogenic pressures, ecological deficits, causes, and rehabilitation/protection measures should be assessed in detail on a water body scale to foster the ongoing process of designing a catchment-specific river basin management plan [30].

Chitosan membrane from crab shell waste with an optimum concentration of 55% has been proven to adsorb Pb (II) metal ions in Tallo river water sample from a concentration of 43 mg/l to 0.187 mg/l. Chitosan membrane adsorption is influenced by a large surface area of the membrane and an enormous adsorption power, and fast adsorption time. Chitosan membrane has a large and hollow pore so that it can bind Pb (II) ions effectively through the mechanism of covalent bonding between amine groups (active site chitosan) and the positive side of Pb (II) ions. The cationic nature of chitosan, which contains amine groups that can bind to metal ions, increases its absorption capacity and selectivity [31]. Thus, the ability of chitosan membrane adsorption of Pb (II) metal ions will reduce the levels of Pb (II) present in Tallo water so that water quality can be maintained and suitable for consumption to meet the fulfillment of clean water for Makassar residents.

3.5. Sustainability of water resource management of Tallo River

During this, efforts to manage and handle water pollution in the Tallo River are partial and conventional. Water management of Tallo River has not been able to meet the acceptable quality of water for consumption and meet the needs of Makassar residents. Sustainability of water resource management of Tallo River requires five essential aspects, namely (i) improvement of water treatment facilities and infrastructure, (ii) application of chitosan membrane technology to adsorb dangerous metal ions, (iii) policy support from the Makassar government to realize sustainable Tallo river water management, (iv) community empowerment, and (v) policy-making for industries to conduct pre-treatments before dumping their waste into Tallo river. Five aspects of river basins, specifically water, ecosystems, socioeconomic development, ability, and data [32]. Furthermore, the sustainability of Tallo River management requires several actions, namely (i) integration of Tallo River management from upstream to downstream, (ii) integration of spatial use management actions along Tallo watershed to maintain the hydrological cycle, and (iii) integration in "green water" and "blue water" management. To realize integrated-water resources management in Tallo watershed, 3 (three) main criteria are needed in its implementation: (i) economic efficiency. Water scarcity for Makassar is significant to be associated with the nature of limited and easily polluted water resources, in the sense that increasing demand makes the economic efficiency of water use critical to be realized, (ii) justice. Water is a basic need of the community, in the sense that all people have the right of access to sufficient water, both in quantity and quality and (iii) environmental and ecological sustainability. Optimizing the use of Tallo river water resources is expected not to sacrifice the interests of future generations towards meeting
the distribution of clean water. The three criteria require several management actions, namely (a) enabling environment in the form of national policies, laws and regulations, and information to stakeholders about water resources management, (b) the role of government institutions and stakeholders at various levels, and (c) instruments management for effective regulation, monitoring and law enforcement. This will require the collaboration of different stakeholders, including water managers, traditional leaders, district to national level government officials, non-governmental organization staff, and researchers [33].

4 Conclusions
The value of the surface area and chitosan membrane adsorption power has provided optimal results with values reaching 765 m$^3$/g, pore volume 1.267 cc/g, and pore diameter 4.7 nm. The large surface area allows considerable membrane adsorption power to adsorb Pb (II) metal ions to neutralize Tallo River quality. Optimization of chitosan membrane concentration in adsorbing Pb (II) metal ions in Tallo River is at an optimum concentration of 55% with Pb (II) metal ions adsorbed at 68.5 mg/g and adsorption capacity of 24.43 mg/g for 25 minutes. Chitosan membrane has been able to adsorb Pb (II) metal ions in Tallo River so that it is suitable for consumption to meet the water needs of Makassar residents.

Water resource management of Tallo River is needed to optimize the distribution of clean water services for Makassar residents. These efforts have been performed through various steps, including requiring wastewater management by preparing water treatment before channeling into water bodies, controlling the utilization of river banks, limiting city activities through zoning arrangements for river benefit areas followed by conservation of land and water resources, construct security building on the left and right sides of the river to prevent erosion and sedimentation, and socialization to the public not to use the river body as a media for garbage disposal. The direction of sustainability is done through 3 (three) main principles, namely the efficient use of water, justice in the right to access to water, sufficient quantity and quality, and environmental sustainability.

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