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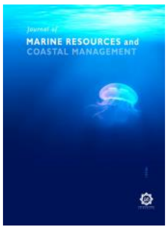

CHITOSAN FROM MILKFISH (*Chanos chanos*) SCALES AND TIGER SHRIMP (*Panaeus monodon*) SHELLS WASTES AS CORROSION INHIBITOR ON ASTM A36 STEEL

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ABSTRACT

Chitosan is synthesized from milkfish (*Chanos chanos*) scales and tiger shrimp (*Panaeus monodon*) shells were used as corrosion inhibitors on ASTM A36 steel. Milkfish scales and tiger shrimp shells wastes is extracted into chitosan through deproteination, demineralization and deacetylation process. The yield of milkfish scales chitosan was 31,81%, while the yield of tiger shrimp chitosan was 67,16%. Corrosion rate testing using weight loss method then calculates inhibition efficiency of each inhibitor. Soaking the test specimens was carried out for four weeks with weight measurements every week. The concentrations used were 0 ppm as negative control, 60 ppm, 500 ppm and 1000 ppm for chitosan inhibitor and 1000 ppm sodium nitrite as positive control. Inhibition efficiency values produced by milkfish scales chitosan, starting from the first week of immersion to the fourth week, were for a concentration of 60 ppm 43.9%; 46.8%; 48.9%; 40.8%; 500 ppm 43.6%; 43.3%; 37.8%; 17.8% and 1000 ppm 37.5%; 44.9%; 39%; 21.8% while for tiger shrimp shells chitosan was at a concentration of 60 ppm 37.6%; 51%; 34.6%; 28.5%; 500 ppm 57.7%; 38.2%; 37.7%; 19.6% and 1000 ppm 48.6%; 41.2%; 37%; 21.3%. Comparison of inhibition efficiency between chitosan from milkfish scales and tiger shrimp shells based on the results of statistical analysis *One Way* ANOVA resulted in a sig >0.05 which indicates a similarity in the efficiency value and the overall average value between chitosan from milkfish scales (38.8%) with tiger shrimp shells (37.75%) has almost the same value, only a difference of 1.05%.

Keywords*:* Milkfish scales chitosan, tiger shrimp shells chitosan, ASTM A36, weight loss, inhibition efficiency

INTRODUCTION

The fishery product processing industry generally produces waste that is not fully utilized. Processing of fishery products such as milkfish products without thorns has processing procedures, such as removing scales, splitting and washing [1]. Fishery products in the form of shrimp also have special processing procedures so that product quality is maintained. Generally, the shrimp to be exported will go through a series of processes before being shipped. The series of processes such as washing, cutting heads, size sorting, stripping, soaking with solutions, freeze and checking in each process [2]. This results in waste in the form of fish scales and shrimp carapace, therefore it is necessary to treat the waste.

ASTM A36 steel (a type of low carbon steel) is one type of material that is often used as raw material for equipment in various industries because of its relatively cheap price and good tensile properties. The main problem in using mild steel is when the steel is in contact with an aqueous environment, especially in the medium (solution) which is acidic [3]. This condition makes the resistance properties of mild steel become weak so it is easy to experience corrosion

Efforts to maximize the use of steel are by preventing or reducing the rate of corrosion. One method to reduce the rate of corrosion is the use of a corrosion inhibitor. Corrosion inhibitors are chemical substances that are added to an environment in order to reduce the corrosion rate of metals [4].

One of the natural ingredients that can be used as a natural inhibitor is chitosan and its derivatives [5]. Fishery waste in the form of fish scales and shrimp shells has many benefits, such as milkfish scales which are a source of chitin and chitosan [6]. The results of research by Harmami, et al. (2019) showed that shrimp shells contain chitosan which can inhibit the corrosion rate of tinplate in 2% NaCl solution [7].

The level consumption of milkfish and tiger shrimp in the community is quite large, resulting in waste that is not used optimally. Milkfish scales and tiger shrimp shells wastes can be used as useful products. In this research, chitosan from milkfish scales and tiger shrimp shells wastes would be syntesized as corrosion inhibitor on ASTM A36 steel in corrosive media in the form of a mixture of 3.5% sodium chloride (NaCl) solution and acetic acid (CH_3COOH) 3%. Chitosan performances as corrosion inhibitor were evaluated by weight loss method.

RESEARCH METHODS

Tools

Bristle brushes, measuring cups, filter paper, digital scales, spatulas, plastic cups, calipers, stationery, hotplates, mattress threads, wooden sticks, beakers glass, salinometers and pH meters were used in this experiment.

Materials

Milkfish scales and tiger shrimp shells waste was collected from Kalanganyar village, Sedati district, Sidoarjo, ASTM A36 steel, sodium hydroxide (NaOH), hydrochloric acid (HCl), aquadest, sodium chloride (NaCl), acetic acid (CH_3COOH), sodium nitrite and wipes.

Research Stages

Stages of this research began with literature studies, preparation of tools and materials, preparation of specimens, preparation of corrosion media, preparation of inhibitors, immersion of specimens, testing of the corrosion rate using the weight loss method, measuring pH and salinity and calculating the efficiency of inhibition.

Specimen Preparation

ASTM A36 steel plate with a thickness of 3 mm cut to a size of 30 mm × 30 mm. The plate is then drilled into the center of the specimen. The hole serves as a place to hang the specimen. Scrubbing the specimen with a bristled brush to remove the coating layer and oxide layer on the specimen surface. In the specimen pickling stage, the specimens are soaked in HCl solution for about 10 minutes, then rinsed with clean water and brushed. The specimens were immersed in NaOH solution, then rinsed with water and dried [8]. The ready specimens are then photographed. Finally, the specimens were weighed using digital scales for initial weight data on the weight loss corrosion rate test [9].

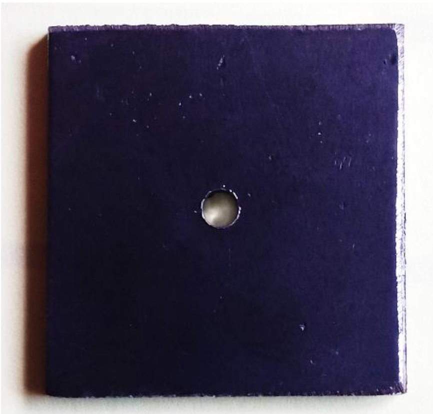

Figure 1. ASTM A36 steel speciment

Preparation of Corrosion Media

The corrosion medium used was a mixture of sodium chloride (NaCl) 3,5% and acetic acid (CH_3COOH) 3%. 3,5% NaCl solution is made from 35 grams of NaCl mixed with aquadest until the volume reaches 1000 ml. 3,5% NaCl solution was put into 200 ml plastic cups each. CH_3COOH is used as a mixture of corrosion media because the chitosan inhibitor can dissolve well in the acid solution acetate (CH_3COOH) 3% [10]. So, to generalize environmental conditions, 12 ml of CH_3COOH 3% was added to all plastic cups in each glass.

Inhibitor Preparations

Natural inhibitor in the form of chitosan is made from milkfish scales and tiger shrimp shells waste. The making of chitosan refers to the manufacturing procedure by Puspawati and Simpen (2010) *in* [11]. The production of chitosan consists of three stages, namely:

1. Deproteination
   Milkfish scale and tiger shrimp shells powder of 80 gram was soaked in 3% NaOH solution dissolved in 1000 ml aquadest, then heated using a hotplate at 80°C for 2 hours while stirring. The solution is filtered and washed to a neutral pH [11].
2. Demineralization
   The result of the deproteination process is added with 1M HCl (1M = 84 ml) dissolved in 1000 ml aquadest, then heated at 75 ° C for 1 hour while stirring. The solution was filtered and the residue obtained was washed to a neutral pH, then dried using an oven at 80°C for 24 hours. This demineralization process produces chitin [11].
3. Deacetylation
   Chitin resulted from the previous process was added with 50% NaOH dissolved in 1000 ml of aquadest, then heated at 75 ° C while stirring. The solution is filtered and washed to a neutral pH. The residue obtained was dried using an oven at 80 ° C for 24 hours. This deacetylation process produces chitosan [11].

Chitosan that has been obtained is then mixed with astetic acid (CH_3COOH) 3%. The variations in the concentration of chitosan inhibitors were 60 ppm, 500 ppm and 1000 ppm. The negative control in this study was without the addition of an inhibitor, while the positive control used sodium nitrite 1000 ppm.

Corrosion Rate Testing with the Weight Loss Method

This experiment used the weight loss method to determine the value of the corrosion rate on ASTM A36 steel. Previously, the specimens were prepared, their surface area was calculate and their initial weight was weighed, immersed in a mixture of NaCl 3,5% and CH_3COOH 3% without and with the addition of an inhibitor for 4 weeks. Weight data collection was carried out once every 1 week of immersion.

Data Analysis

Percentage of chitin and chitosan analysis was calculated using a formula as follow [10]:

$$\text{Percentages (\%)} = \frac{\text{weight of chitin/chitosan}}{\text{weight of sample}} \text{x}100\%$$

Data that has been obtained in the form of the surface area of the specimen and the lost mass then the corrosion rate is calculated using the weight loss calculation formula which refers to [9] :

$$CR = K\frac{W}{DAT}$$

Where :
CR = corrosion rate (mmpy)
W = mass loss (g)
A = surface area of specimen (cm^2)
T = time of exposure (hours)

D = density of specimen (g/cm^3)
K = a constant (8,76 × 10^4)

Percentage of inhibition efficiency with the weight loss method can be calculated by the following formula [12]:

$$\%EI = \frac{(CRuninhibited - CRinhibited)}{CRuninhibited} x100\%$$

Where :
%EI = inhibition efficiency (%)
CRuninhibited = corrosion rate on corrosive media without inhibitor
CRinhibited = corrosion rate in corrosive media with the addition of inhibitors

RESULTS AND DISCUSSIONS

Chitosan from Milkfish Scales and Tiger Shrimp Shells

Chitosan in this research was utilized as an inhibitor of ASTM A36 steel in a mixture of NaCl 3,5% and CH_3COOH 3% solutions. The synthesis of chitosan was carried out based on the research procedures of Puspawati and Simpen (2010) *in* [11]. Powder of milkfish scales (*Chanos chanos*) and tiger shrimp shells (*Panaeus monodon*) were extracted into chitin through deproteination and demineralization processes. The next process is that chitin is synthesized into chitosan through deastelization process. Chitosan from milkfish scales is a brownish white powder. Black tiger shrimp shell chitosan is a light brown powder with a slightly orange color. The chitosan powder from milkfish scales is smaller than the chitosan produced from tiger shrimp shells.

Based on the data in **Table 1**, it is known that the percentage of chitin from tiger shrimp shells has a greater value than chitin from milkfish scales. This is because milkfish scales contain 45,07% protein [13] while tiger shrimp shells contain 14,85% protein [14]. The protein content in the large milkfish scales makes the initial weight decrease quite a lot because in the deproteination process, the protein contained in the milkfish scales will dissolve in the base. The initial weight decreased quite a lot, making the percentage of chitin yield has a smaller value than the chitin yield from tiger shrimp shells.

Table 1 Percentage of chitin and chitosan from milkfish scales and tiger shrimp shells wastes.

	Milkfish Scales	Tiger Shrimp Shells
Chitin (%)	55	83,75
Chitosan (%)	31,81	67,16

Deacetylation process using a strong base and heat resulting in the loss of an acetyl groups on the causes of chitin chitosan charged positive so that it dissolves in acidic solutions such as acetic acid. The yield percentage of chitosan from tiger shrimp shells was 67,16%, this result was in accordance with the research that the previously, the percentage of shrimp shell chitosan was 67,08% [10].

The Effect of Addition of Inhibitors on the Corrosion Rate of ASTM A36 Steel

Average data of the corrosion parameters on ASTM A36 steel in a mixture of NaCl 3,5% and CH_3COOH 3% solutions can be seen in Table 2, 3, 4 and 5. Based on **Table 2** it can be seen It is known that soaking 1 week the smallest corrosion rate value is in the addition of chitosan from tiger shrimp shell with a concentration of 500 ppm of 0,21482 mmpy. Soaking 2 weeks, the lowest corrosion rate value is in the addition of chitosan from tiger shrimp shell with a concentration of 60 ppm of 0,16881 mmpy. Soaking 3 weeks, value lowest corrosion rate was in the addition of 60 ppm of chitosan from milkfish scales of 0,12119 mmpy. Soaking 4 weeks, the lowest corrosion rate was the addition of 60 ppm of chitosan from milkfish scales with a value of 0.10995 mmpy.

Figure 2 Specimen with chitosan inhibitor after soaking for one week

The inhibitor of chitosan from tiger shrimp skin and chitosan from milkfish scales with a concentration of 60 ppm and 500 ppm had an average corrosion rate lower than the corrosion rate value of chitosan with a concentration of 1000 ppm. This shows that chitosan from milkfish scales and chitosan from tiger shrimp shells have the optimum concentration to inhibit the corrosion rate, so that when the inhibitor added to the corrosive medium exceeds the optimum concentration, the inhibitor will experience saturation or may experience the release of corrosion inhibitors [15].

Data in **Table 2** shows that the value of the corrosion rate on ASTM A36 steel with a salinity of 27,16 ppt is 0,28499 mmpy, a salinity of 28,70 ppt is 0,31470 mmpy and a salinity of 30,20 ppt is 0,50804 mmpy. It can be seen that the greater the salinity of the corrosion media, getting greater to the value of the corrosion rate [16].

Based on **Table 3** it can be seen that the media without the addition of an inhibitor has a pH of 5,43 with a corrosion rate value of 0,34174 mmpy, in media with the addition of a sodium nitrit concentration is 1000 ppm has a pH of 4,87 with a corrosion rate value of 0,18774 mmpy, in media with the addition of chitosan from milkfish scales concentration is 60 ppm has a pH of 4,76 with a corrosion rate value of 0,18173 mmpy and in media with the addition of chitosan from tiger shrimp shells concentration is 60 ppm has a pH of 4,67 with a corrosion rate value of 0,16881 mmpy. These results are inversely proportional to the statement of Sidiq (2013) which states that the corrosion rate will increase at pH <7 and pH> 13 [17].

This difference is because chitosan can work at an acidic pH, so with a lower pH than corrosive media without the addition of an inhibitor, the value of the corrosion rate produced by the addition of a chitosan inhibitor will have a smaller corrosion rate value. This is in accordance with the statement of Erna, et al. (2011) which states that at pH 6 chitosan begins to dissolve, making it easier for the functional groups in the form of –OH and $–NH_2$ to bind to the steel surface [12].

Apart from pH and salinity, dissolved solids are also a factor affecting the corrosion rate. This research used corrosive media in the form of a mixture of sodium chloride (NaCl) 3,5% and acetic acid (CH_3COOH) 3% solutions. Chloride (Cl) attacks mild steel and stainless steel layers. This solid chloride (Cl) causes pitting corrosion, crevice corrosion and can also cause the breakdown of alloys [17].

Table 2 Average data of corrosion parameters on ASTM A36 steel in a mixture of NaCl 3,5% and CH_3COOH 3% solutions soaking 1 week

1 Week					
Inhibitor	**Conc. (ppm)**	**(W-W')(g)**	**CR (mmpy)**	**Sal.**	**pH**
Control negative*	0	0,1667	0,50804	30,20	5,63
Chitosan milkfish scales	60	0,0933	0,28499	27,16	5,67
	500	0,0920	0,28642	28,36	5,67
	1000	0,1020	0,31747	28,70	5,63
Chitosan tiger shrimp shells	60	0,1026	0,31684	28,10	5,63
	500	0,0700	0,21482	28,16	5,67
	1000	0,0850	0,26118	29,16	5,67
Control positive**	1000	0,1046	0,31878	29,53	5,63

Table 3 Average data of corrosion parameters on ASTM A36 steel in a mixture of NaCl 3,5% and CH_3COOH 3% solutions soaking 2 weeks

2 Weeks					
Inhibitor	**Conc. (ppm)**	**(W-W')(g)**	**CR (mmpy)**	**Sal.**	**pH**
Control negative*	0	0,2227	0,34174	33,13	5,43
Chitosan milkfish scales	60	0,1173	0,18173	27,63	4,76
	500	0,1260	0,19378	29,47	4,76
	1000	0,1207	0,18816	29,23	4,87
Chitosan tiger shrimp shells	60	0,1093	0,16881	27,17	4,67
	500	0,1360	0,21111	29,67	5,17
	1000	0,1300	0,20107	30,67	5,30
Control positive**	1000	0,1227	0,18774	30,67	4,87

Table 4 Average data of corrosion parameters on ASTM A36 steel in a mixture of NaCl 3,5% and CH_3COOH 3% solutions soaking 3 weeks

3 Weeks					
Inhibitor	**Conc. (ppm)**	**(W-W')(g)**	**CR (mmpy)**	**Sal.**	**pH**
Control negative*	0	0,2327	0,23739	33,87	5,13
Chitosan milkfish scales	60	0,1193	0,12119	28,10	5,17
	500	0,1433	0,14768	29,90	5,03
	1000	0,1407	0,14480	30,30	5,07
Chitosan tiger shrimp shells	60	0,1513	0,15528	27,70	4,93
	500	0,1447	0,14784	31,00	4,80
	1000	0,1440	0,14957	31,20	5,07
Control positive**	1000	0,1467	0,14996	31,10	4,90

Inhibition Efficiency of Chitosan from MilkfishScales and Chitosan from Tiger Shrimp Shells

The value inhibition efficiency of chitosan from milkfish scales, chitosan from tiger shrimp shells and sodium nitrit starting from 1 week to 4 weeks of immersion can be seen in Table 6. The optimum inhibition efficiency at 1 week of immersion using chitosan from tiger shrimp shells concentration is 500 ppm with a value of 57,7%. Soaking for 2 weeks the optimum inhibition efficiency using chitosan from tiger shrimp shells with a concentration of 60 ppm with a value of 51%. Soaking for 3 weeks the optimum inhibition efficiency using chitosan from milkfish scales concentration is 60 ppm with a value of 48,9%. Soaking 4 weeks, optimum inhibition efficiency using chitosan from milkfish scales concentration is 60 ppm with a value of 40,8%.

Table 5 Average data of corrosion parameters on ASTM A36 steel in a mixture of NaCl 3,5% and CH_3COOH 3% solutions soaking 4 weeks

4 Weeks					
Inhibitor	**Conc. (ppm)**	**(W-W')(g)**	**CR (mmpy)**	**Sal.**	**pH**
Control negative*	0	0,2433	0,18568	32,50	4,83
Chitosan milkfish scales	60	0,1433	0,10995	27,17	4,93
	500	0,1967	0,15257	31,10	4,57
	1000	0,1873	0,14511	30,70	4,43
Chitosan tiger shrimp shells	60	0,1720	0,13277	28,10	4,60
	500	0,1940	0,14922	31,20	4,70
	1000	0,1893	0,14610	33,30	4,50
Control positive**	1000	0,1713	0,13222	32,03	4,83

Control negative* = Without inhibitor
Control positive** = Sodium nitrit 1000 ppm

Inhibition efficiency in chitosan from milkfish scales with a concentration of 500 ppm and 1000 ppm has a lower value than the inhibition efficiency of a concentration of 60 ppm. This is in accordance with the research results of Hidayatullah, et al. (2019) which states that the highest inhibition efficiency value is produced by an inhibitor with a concentration of 100 ppm while for a concentration of 200 ppm, 300 ppm and 400 ppm continues to experience a decrease in efficiency. The inhibition efficiency is in accordance with the corrosion rate (mm / year) value of ASTM A36 which has the lowest corrosion rate value produced by specimens with an inhibitor concentration of 100 ppm [18].

The results in **Table 6** show that the inhibition efficiency of chitosan from tiger shrimp shells increased and reached its optimum at a concentration of 500 ppm but decreased the efficiency value at a concentration of 1000 ppm. This is in accordance with the statement of Harmami, et al. (2019) which states that the addition of Water Soluble Chitosan (WSC) of 10-1300 mg / L can block the surface where corrosion occurs so that it inhibits the metal dissolving process while the addition of WSC of 1500 mg /L occurs diffusion of inhibitors into the solution so that inhibition efficiency decreases [7].

Sodium nitrite is one type of synthesis inhibitor. Sodium nitrite in this research was used as a positive control. According to Nikitasari, et al. (2014), sodium nitrite is a very effective inhibitor to inhibit the corrosion of steel reinforcement which is contaminated with chloride [19]. The data from Table 6 shows that the efficiency of the sodium nitrite with a concentration of 1000 ppm has a lower efficiency value when compared to the chitosan from milkfish scales and chitosan from tiger shrimp shells with a concentration of 1000 ppm within 1 week to 3 weeks of soaking time. This is because the corrosive solutions used in this research are acidic and chitosan inhibitors can work better at low pH or acidic ones [12] while sodium nitrite inhibitors can work well in neutral or alkaline conditions [19].

Table 6 Percentage of inhibition efficiency from 1 week to immersion to 4 weeks

Inhibitor	**Conc. (ppm)**	**Inhibition Efficiency (%)**			
		Soaking Week			
		1	**2**	**3**	**4**
Chitosan milkfish scales	60	43,9	46,8	48,9	40,8
	500	43,6	43,3	37,8	17,8
	1000	37,5	44,9	39	21,8
Chitosan tiger shrimp shells	60	37,6	51	34,6	28,5
	500	57,7	38,2	37,7	19,6
	1000	48,6	41,2	37	21,3
Control (+) positive**	1000	37,3	45,1	36,8	28,8

Control positive** = Sodium nitrit 1000 ppm

The inhibition efficiency comparison curve can be seen in **Fig. 3**. The curve shows that the inhibition efficiency value of chitosan from milkfish scales and tiger shrimp shells has different values, although the percentage difference is not significant. This is because the ash content in the chitosan from milkfish scales and chitosan from tiger shrimp shells is not much different. Chitosan from milkfish scales contains an ash content of 1,15% [6] while chitosan from tiger shrimp shells contains an ash content of 0,99% [20]. The ash content contained in chitosan from milkfish scales and tiger shrimp shells has a small value so that the chitosan has the same efficiency value or does not differ much in inhibiting the corrosion rate of the test specimens. [7].

The insignificant comparison of the inhibition efficiency between milkfish scales chitosan and tiger shrimp shells, which is not too significant, can also be seen in the results of statistical analysis using the *One Way* ANOVA method. The results of the homogeneity test showed that the sig value of milkfish scales chitosan was 0.263 while the tiger shrimp shell chitosan sig value was 0.862. The value of sig> α (0.05), it can be concluded that the chitosan of milkfish scales and tiger shrimp shells have the same or homogeneous variant values so that the prerequisites for using ANOVA are fulfilled[21]. ANOVA test results showed that the sig value of milkfish scales chitosan was 0.303, while the chitosan value of tiger shrimp shell chitosan was 0.989. The sig value> 0.05 so it can be concluded that there is an average equation between the inhibition efficiency value of chitosan from milkfish scales and chitosan from tiger shrimp shells with a concentration of 60 ppm, 500 ppm and 1000 ppm [21]. Inhibition efficiency for 4 (four) weeks produced chitosan from milkfish scales is 60 ppm is 45.1%, concentration of 500 ppm 36.6% and 1000 ppm 35.8% with the total average value of the overall concentration is 38.8%. The average value for 60 ppm chitosan from tiger shrimp was 37.9%, concentration of 500 ppm 38.3% and 1000 ppm 37% with the overall average total concentration of 37.75%. So, it can be concluded that when viewed from the overall average value of the concentration between chitosan from milkfish scales and tiger shrimp shells, it has almost the same value, only a difference of 1.05%.

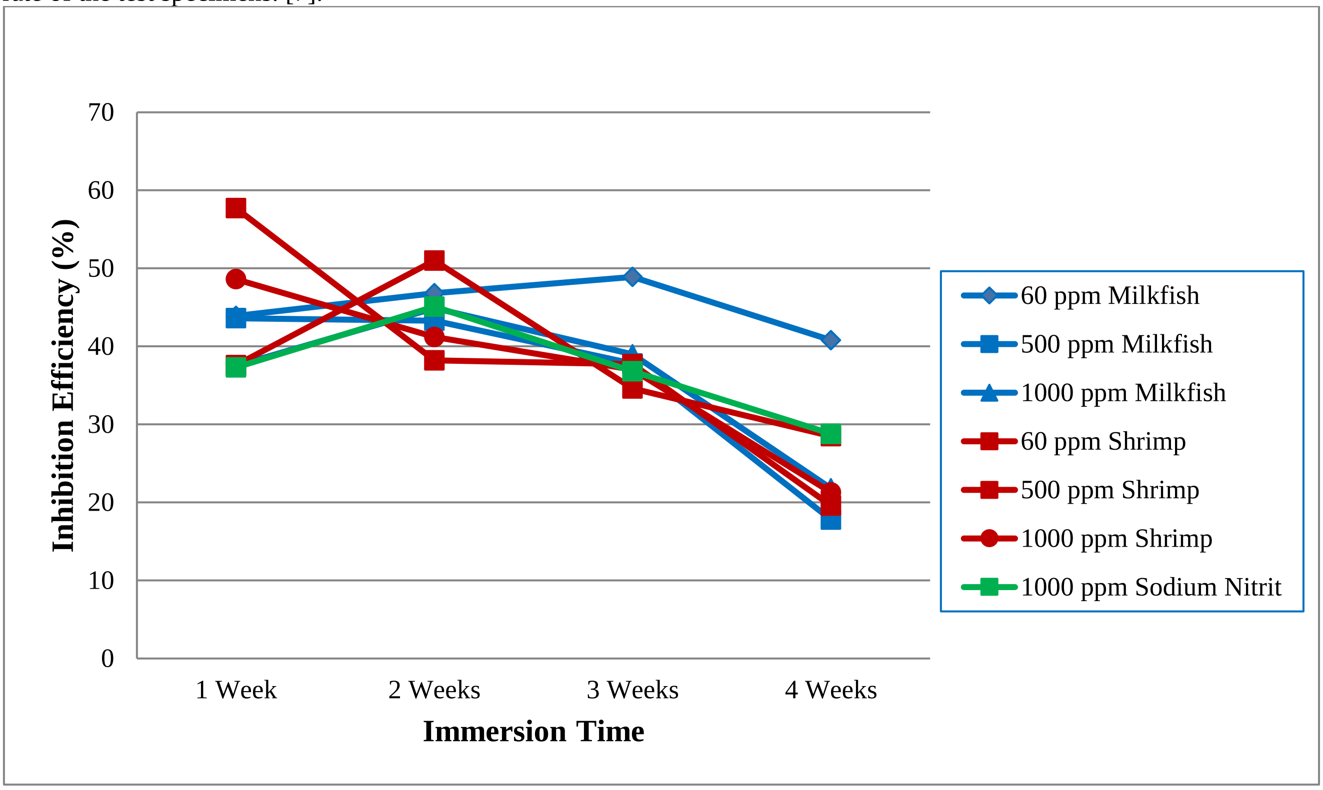

Figure 3. Inhibition Efficiency of Milkfish Scales Chitosan, Tiger Shrimp Shell Chitosan and Sodium Nitrite

CONCLUSIONS

Inhibition efficiency of chitosan from milkfish (*Chanos chanos*) scales as an inhibitor of corrosion rate on ASTM A36 steel consecutively starting from the first week of immersion to the fourth week is for a concentration of 60 ppm 43.9%; 46.8%; 48.9%; 40.8%; concentration of 500 ppm 43.6%; 43.3%; 37.8%; 17.8% and a concentration of 1000 ppm 37.5%; 44.9%; 39%; 21.8%. Meanwhile inhibition efficiency of chitosan from tiger shrimp (*Panaeus monodon*) shells as a corrosion rate inhibitor on ASTM A36 steel consecutively starting from the first week of immersion to the fourth week is for a concentration of 60 ppm 37.6%; 51%; 34.6%; 28.5%; concentration of 500 ppm 57.7%; 38.2%; 37.7%; 19.6% and a concentration of 1000 ppm 48.6%; 41.2%; 37%; 21.3%. The data shows inhibition efficiency between chitosan from milkfish scales and tiger shrimp shells has almost the same value, which is shows the similarity in the efficiency value and from the average value of the overall concentration from the first week to the fourth week between chitosan from milkfish scales (38.8%) and tiger shrimp shells (37.75%) has almost the same value, only the difference 1.05%.

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