

Inquiry-Based Worksheet On The Utilization Of Pectin From Lemon Peel Waste As Corrosion Inhibitors To Support Student Understanding In Electrochemistry Concept

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Abstract

In order to improve the quality of education, construction of a new, alternative experiment worksheet in Basic Chemistry course is needed. Electrochemistry concept, especially corrosion was the main concern of this research because this concept is considered difficult by first year college student. The aim of this research was to produce inquiry-based worksheet on utilization of pectin as corrosion inhibitor. Pectin was utilized in this worksheet because it has

been known to possess corrosion inhibition properties and environmental-friendly. The method used was 2D (define and design) research and development model. The steps of this research was optimization of experimental procedure, developing the design of the inquiry-based worksheets, and conducting limited test to ten General Chemistry II course students. Pectin was obtained through lemon peel extraction using HCl and ethanol. The rate of corrosion measured on iron plates in HCl 1 M aqueous media. Inhibition efficiency was calculated using weight loss method. Optimum pectin inhibition efficiency was 78%. Validation test of the development of worksheet procedure showed that the worksheet was valid and feasible with r value = 0.78 and eligibility percentage = 79.16%. This findings showed that the worksheet is applicable in teaching chemistry on corrosion topic.

Key Words: Inquiry; worksheet; pectin; lemon peel waste; corrosion inhibitor.

1 Introduction

An experiment can be carried out more effectively with the use of experiment worksheet. Experiment worksheet that is commonly and widely used is experiment worksheet which stated step-by-step procedure that has to be done by students. Wenning reveals that this kind worksheet was not able to fulfill the scientific thinking skills of most students because it only contains detailed procedures so that the students only follow the procedures in the worksheet without thinking about the reason (1). Therefore, a worksheet that can improve students' scientific thinking skill is needed. According to Maryati et al., students scientific thinking skill can be developed through an inquiry-based worksheet (2). Joyce and Weil reveal that the core of inquiry learning is students involvement in identifying and designing the methods to solve a problem (3). Inquiry-based learning processes include formulating problems, designing experiments, collecting data, analyzing data, and making conclusions. Brown et al stated that an inquiry-based worksheet can improve learners' motivation, because learners can find problems themselves and solve the problems independently (4).

Inquiry based worksheet can be applied to an experiment about corrosion. Corrosion is an example of contextual event related to the concept of redox reaction and electrochemistry in Basic Chemistry course. Corrosion is an environmental problem often encountered in everyday life that has to be resolved. Corrosion mostly occurs on the surface of iron-based objects or components because of the negative reduction potential. This metal is still used in large quantity because of its high abundance in Earth crust, cheap metal processing, and easy fabrication. Nasution, et al. said that the impacts caused by corrosion will affect the environment such as soil pollution (5).

Corrosion also can be prevented temporarily through physical or chemical methods. Corrosion rate can be reduced by the use of corrosion inhibitor. Corrosion inhibitor can be divided into two types: inorganic inhibitor and organic inhibitor. Examples of inorganic inhibitor include arsenates, chromates, silicates, and phosphates that is harmful to the environment. The use of natural organic materials as corrosion inhibitor materials is preferred because it is environmental-friendly. Effective organic inhibitor usually contains nitrogen, sulfur, oxygen, and phosphorus atoms in their molecular structures (6). In addition, organic compounds containing electronegative atom and electron in double bond or triple bond are also good inhibitors. The action mechanism of organic inhibitor is by adsorption on metal surface. The use of natural polymer structures derived from leaf extracts, seeds, or fruit peel as a natural corrosion inhibitor is good and safe to use. One of the organic compounds that can be used as a corrosion inhibitor is pectin (7).

Pectin contains a carboxylic functional group (-COOH) and carboxymethyl (-COOCH₃) which is an active compound to prevent corrosion (8). Pectin is a heterosaccharide polymer obtained from cellular plant cell walls. Pectin can be obtained from apples, bananas, oranges, lemons, carrot, and cacao peels. Utilization of lemon peel waste as corrosion inhibitor is a great potential for pectin production. According to Gregory, pectin content in the lemon peel is 35% (9). In this study, lemon peel waste is processed to produce pectin which will be used as a corrosion inhibitor. The inhibition efficiency of pectin against corrosion rate was calculated. Previous research by Fares et al on the use of pectin from orange peel as a corrosion inhibitor of aluminum metal using chloride acid solution

medium as a corrosive environment gives the conclusion that pectin is an effective corrosion inhibitor (10).

The aim of this research was to optimize simple experimental procedure to be applied in an inquiry-based worksheet. The experimental procedure is expected to be an alternative for Basic Chemistry teaching activity in concept about corrosion. Learning through inquiry experiment is expected to enhance students' understanding of corrosion concepts and also to open up a broad knowledge of corrosion that has been a problem of the environment. At the best of our knowledge, there are no published paper about inquiry-based worksheet in the topic of corrosion inhibition by pectin.

2 Methodology/Materials

The method used in this research is research and development method. Sugiyono and Sukmadinata suggests that research and development methods are research methods used to produce a particular product and test the effectiveness of the product(11, 12). Research and development method in this study is carried out with 2D stages: define and design. Define stage is performed by analyzing basic chemistry concept which is considered difficult, but related to everyday life (redox reaction and electrochemistry concept, especially about corrosion), analyzing relevant journals on corrosion such as prevention of corrosion with inhibitors, pectin compounds as corrosion inhibitors, and procedures of pectin isolation from lemon peel, and analysis of inquiry-based learning process. Design stage is performed by conducting preliminary tests in the laboratory, then compiling inquiry-based worksheet which is further validated by content expert. Further improvement is done according to suggestions and input validation results The subject of this research is the expert lecturer as validator of the resulting worksheet and 10 college students who have taken Basic Chemistry as subjects for the worksheet limited test. Since the worksheet will be used by the student, it must be tested in advance whether the worksheet is appropriate to use or not. The competent authority to test the worksheet in this case is a chemistry expert.

The instrument used in this study consists of the student work-

sheet supplemented with feasibility test form, the table of determination of concentration and the optimum time of pectin immersion as iron corrosion inhibitor, and the tools and materials used in this research.

2.1 Worksheet

Inquiry-based worksheet is prepared based on the analysis of content of Basic Chemistry course, which is considered difficult but related to daily life. Worksheet is arranged in accordance with the stages of inquiry proposed by Trianto and Maryati, et al, consisting of the stage of formulating the problem, making hypotheses, designing experiments, conducting experiments, analyzing data, and making conclusions (2, 13).

2.2 Feasibility and Validity Test Form

Instruments for the feasibility test form used in this study are questionnaires. The feasibility test form is used to test the feasibility of the worksheet that has been prepared in accordance with the procedures generated in this study. The worksheet used in the use of pectin as an iron corrosion inhibitor is an inquiry-based worksheet. According to Suharsimi, the feasibility test form is using rating scale, with four alternative points: very good = 4; good = 3; not really good = 2; and not good = 1 (14). After getting the answers, the data is processed into a percentage with the formula in equation (1). The feasibility test result is concluded according to the criteria in table 1.

Percentage of response =

$$\frac{f}{n} \times 100\% \quad (1)$$

With f = frequency response, and n = number of respondents.

Table 1. Feasibility criteria based on percentage of worksheet response (15)

Percentage (%)	Qualification	Conclusion
90-100	Very feasible	Worksheet is ready for use by student as a tool in conducting experiments
80-89	Feasible	Worksheet is ready for use by student as a tool in conducting experiments
70-79	Fairly feasible	Worksheet can be used by enhancing the lacking part according to the advice of the expert lecturers
60-69	Less feasible	Revise and re-examine the resulting worksheet, examine the drawbacks of the worksheet, and revise it.
<60	Very inadequate	The worksheet failed to pass the feasibility test, cannot be used and should do a massive revision

In the validation test, data from questionnaires are then analyzed to calculate the validity of the worksheet. The test can be done by comparing the feasibility value (r) with the critical value of r (rcritical) according Sugiono (11). The calculations carried out using correlation technique, mathematically the formula used can be seen in equation (2).

$$r = \frac{x}{N.n} \tag{2}$$

where :

r = feasibility value

x = point/score of respondents answer

N = number of items

n = number of respondents

According to Masrun, correlation techniques to determine the validity of items is the most widely used techniques. Also, items that have a high correlation indicates that the item has a high validity (11). Minimum requirement to be concluded valid is $r = r_{critical} = 0.3$. If r value is less 0.3 the item is concluded invalid. The r value interpretation can be seen in table 2.

Table 2. Interpretation of Eligibility Values (11)

No.	The value of r	Interpretation
1	0.8 – 1	Very high
2	0.6 – 0.8	High
3	0.4 – 0.6	Medium
4	0.2 – 0.4	Low
5	0.0 – 0.2	Very low

2.3 Experimental procedure for measuring corrosion rate

Procedure for measuring corrosion rate requires some tools and materials such as iron plates, glass bottles, sandpaper, pipette, oven, thread, graduated cylinder, analytical balance, spatulas, graduated

flasks, and watch glass. The data obtained from corrosion rate testing involves iron initial weight and iron final weight after soaking in corrosive solution. The iron reduced weight will be used to calculate corrosion rate and efficiency of inhibitor (inhibition efficiency).

3 Results and Findings

3.1 Description of The Worksheet

The inquiry-based worksheet on the utilization of pectin as corrosion inhibitor was successfully compiled. This worksheet contains six phases of inquiry, involves: 1) presenting problems, 2) making hypotheses, 3) designing experiments, 4) experimenting, 5) analyzing data, and 6) drawing conclusions. Front page of the worksheet contains the purpose of experiment and instructions about how to use the worksheet. The purpose of the experiment was to determine corrosion rate of iron using weight loss method in corrosive media with and without pectin inhibitor. In the instruction section, it was stated that students can use books and information from the internet to collect the required data, students have to work and discuss the given problems in groups, and students were requested to read the provided text about corrosion.

The students read the provided text for around five minutes. Trianto stated that inquiry learning begins when questions or problems are raised through observations in text so as to encourage students to think critically and analytically (16). The text provided in the worksheet was titled Pectin from Lemon Peel as Corrosion Inhibitor. First and second paragraph in the text explains about corrosion, factors affecting corrosion, methods used to prevent corrosion that leads to a corrosion inhibitor, and characteristics of organic corrosion inhibitor as well as many advantages of organic inhibitors. Third paragraph introducing pectin as one of many alternatives for corrosion inhibitor, explains what is pectin, source of pectin, and how to isolate pectin. This paragraph also explains the method to investigate inhibition properties of pectin. Fourth (last) paragraph explains about mathematical formula used to calculate corrosion rate and inhibition efficiency using data obtained from method in third paragraph.

Inquiry phases are stated implicitly in the worksheet questions

that follows after the provided text. First and second phases of inquiry learning (presenting problems and making hypotheses) represented by questions that ask students to imply the problem that is described in the text as well as possible solutions to the problem and ask students to suggest some hypotheses for the possible solution. Students are then challenged to design an experiments to test their hypotheses about the inhibition properties of pectin to iron corrosion. In this stage, students are free to decide their desired variables in the experiments that they want to perform with an instructor consent and guidance in the process. Fourth phase is performing experiment. In this phase, the students performed the experiment designed by their group in the third stage. The data obtained from the experiment then analyzed by each student group in fifth stage. In the last stage (drawing conclusions), students are encouraged to describe their findings and conclusions to their performed experiments.

3.2 Worksheet Feasibility and Validation Test Result

The worksheet that has been prepared was validated by three expert lecturers. The validation test results and percentage of feasibility of this worksheet are presented in table 3 and table 4.

Table 3 Recapitulation of Validation Test Results from Three Expert Lecturers

No.	Statement	r value	r critical	Validity
1	The sentence used in the inquiry-based worksheet is easily understood	0,75	0,3	Valid
2	Questions used on the worksheets are accordance with the phases of inquiry	0,83	0,3	Valid
3	The provided text about corrosion events and its prevention efforts can assist students in arranging the experiment procedure	0,66	0,3	Valid
4	The provided text about corrosion events and prevention efforts can assist students in answering questions	0,83	0,3	Valid
5	Questions given on the worksheet assist students in digging information	0,83	0,3	Valid
6	The experiment on the utilization of pectin from lemon peel as a corrosion inhibitor can be done easily	0,75	0,3	Valid
7	The tool used in the experiment is easily obtained	0,75	0,3	Valid
8	The material used in the experiment is easily obtained	0,75	0,3	Valid
9	The concept relates to daily life	0,91	0,3	Valid
10	Worksheet is in accordance with curriculum and basic competence	0,83	0,3	Valid
Total		78	30	
Average		0,78	0,3	

Based on the validation test results in table 3, the constructed worksheet reached an average of rvalue of 0.78. When compared with the minimum requirement of the feasibility test result, it was found rvalue is higher than rcritical. Therefore, it was concluded that the worksheet has fulfilled the requirement and declared as valid. All statements in the worksheet validation form were concluded as valid. Each statement has a different rvalue. The highest value of r was in statement number nine (0.91), while the lowest value of r was in statement number three (0.66). The lowest value in statement number three means that in this third statement some reviewers generally understand the procedure presented in the provided text and some consider it unclear. Therefore, based on the results of worksheet validation test, we have made some improvements to the worksheet.

Table 4 Percentage of Feasibility Test Results

Validator	Percent of Feasibility	Judgement	Suggestion for improvement
1	82,5%	The worksheet is ready for use by students as a tool in conducting experiments	- List reference journals that refer to the experiment - Text can be improved by adding general experimental procedures
2	75%	The worksheet can be used by enhancing the lacking aspects according to the suggestion of the experts, and not making large-scale revisions	Enhance the text and questions
3	80%	Lemon skin pectin utilization as iron corrosion inhibitor worksheet is ready for use by students as a tool in conducting experiments	-Improve the text and questions -List journal reference in the text -Improve experiment design by creating a template

Based on table 4, the average percentage of feasibility test results by expert lecturers is 79.16% with sufficient qualification. The worksheet can be used by enhancement according to the advice of the expert lecturers, and not make a massive revision.

3.3 Results of The Procedure Optimization

The first step in the procedure optimization conducted was pectin isolation from lemon peel. After enough quantity of pectin for procedure optimization and for limited test by 10 students is reached, second procedure of the utilization of pectin as corrosion inhibitor can be carried out. The aim of the procedure optimization is to obtain optimum soaking time of iron to corrosive media and to obtain optimum concentration of inhibitor. The procedures and data from

this procedure optimization will be used as reference procedure for designing experiment in inquiry phase.

Pectin used in this research was isolated from lemon peel waste obtained from the traditional market. Pectin was isolated from lemon peel by using hydrochloric acid and ethanol. The isolated pectin is then applied as an iron corrosion inhibitor. The next step of this research is determination of optimum concentration of corrosion inhibitor, in this case corrosion in iron. The corrosion inhibitor used was pectin which was directly dissolved in corrosion medium HCl 1 M. Pectin concentration used was 2.0 g / L, 4.0 g / L and 6.0 g / L with each concentrated tested three times (triplo). The optimum concentration can be known from the rate of corrosion based on the variation of concentration used. Determination of the corrosion rate is done by weight loss method. The weight loss method involves the process of cleaning the metal used, weighing the metal before it is immersed into the corrosion medium, cleaning the metal after corrosion, and weighing the final weight of the metal after it is corroded. The metals used in this study were iron plates with size 1.6 4 0.1 cm, hung in a corrosion medium using a thread for 2 hours at room temperature, both without and with the addition of a corrosion inhibitor. The weight losses of iron are presented in table 5. Iron immersion with the addition of inhibitor, with variation of concentration of inhibitor in corrosion media, obtained the result that the inhibitor concentration has an effect on the weight loss of iron as in table 5.

Table 5 Reduction of Iron Mass

Inhibitor Concentration	Test No.	Iron Mass (gram)		
		W ₀	W _a	W
Blank (0 g/L)	I	2.0211	2.0179	0.0032
	II	2.0301	2.0270	0.0031
	III	2.0290	2.0245	0.0045
2 g/L	I	1.9977	1.9949	0.0028
	II	1.9966	1.9943	0.0023
	III	1.9995	1.9979	0.0016
4 g/L	I	1.9827	1.9819	0.0008
	II	1.9941	1.9934	0.0007
	III	1.9952	1.9945	0.0007
6 g/L	I	1.9607	1.9598	0.0009
	II	1.9630	1.9622	0.0008
	III	1.9629	1.9621	0.0008

W₀ = Initial weight of iron before immersion in corrosion media
 W_a = Final weight of iron after iron Immersion
 W = Weight loss of iron (W₀-W_a)

Based on table 5, the addition of corrosion inhibitor with various concentrations affects the weight of iron lost in the final weighing. The more concentration of inhibitor added in the corrosion medium the less weight of iron lost, but on the addition of corrosion inhibitor as much as 6 g / L the lost weight of iron slightly increased from the inhibitor concentration of 4 g/L. However, this insignificant increase can be considered not to occur because of the tool precision (0.0001 gram). Weight reduction of iron is caused by iron oxidation to iron (II) ions (17).

Corrosion media used in this study is HCl. HCl is a powerful electrolyte substance that can corrode iron because chloride ions can react with ferric metals to form iron (II) chloride compounds. Acidic media will accelerate the rate of iron corrosion, this is due to the influence of environmental pH which is the degree of acidity of the environment that indicates the concentration of H⁺ in the environment. Based on table 5, the addition of corrosion inhibitor with various concentrations affects the weight of iron lost when the final weighing. The more concentration of inhibitor added in the corrosion medium the less weight of iron lost, but on the addition of corrosion inhibitor as much as 6 g/L the lost weight of iron slightly increased. In this study, to coat the entire surface of the metal apparently requires a concentration of inhibitor of 4 g/L to protect the metal under optimum conditions. Corrosion rate is calculated and plotted against inhibitor concentration in Figure 1.

The data in the figure 1 shows that the corrosion rate decreases with the addition of pectin inhibitors into the corrosion medium, compared with the blank solution (without the addition of the inhibitor). At a concentration of 4 g/L, rate of corrosion is at minimum value. However, in addition to 6 g/L inhibitor concentration the corrosion rate slightly increased. Based on the results of this study it can be concluded that the addition of pectin inhibitor of 4 g/L is the optimum concentration. The efficiency of inhibitor can be seen in figure 2.

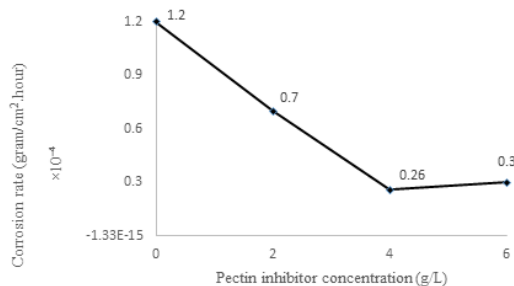


Figure 1 Plot of inhibitor concentration vs corrosion rate with two hours immersion time

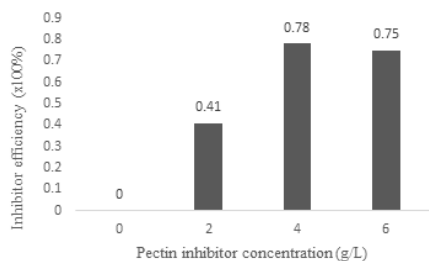


Figure 2 Effect of inhibitor concentration to inhibition efficiency

Based on the data on figure 2, the highest efficiency of pectin inhibitor in reducing the corrosion rate is at 4 g/L with 78% inhibition efficiency and the lowest efficiency is in the addition of 2 g/L inhibitor with only 41% efficiency. The next step in this research is determining the optimum immersion time in 1M HCl corrosive media by using the same inhibitor concentration of 4 g/L. The immersion time varied to 1, 2, and 3 hours, each without and with the addition of inhibitor. The optimum time is obtained from the smallest corrosion rate. The effect of immersion time to corrosion rate is presented in figure 3. Based on the data on the graph, the highest inhibition efficiency was achieved at two hours immersion time, with 78% efficiency. Thus, the optimum immersion time in this study was two hours.

For comparison, previous studies by Fiori et al. with pectin isolated from orange peel as a corrosion inhibitor of light steel resulted in an optimum concentration of 2 g/L (7). Light steel has less iron content than iron in pure state. This is because there are other

substances in light steel such as phosphorus, silicon, aluminum, manganese, and carbon. In the use of light steel, inhibitor needed coat metals will be required in less quantity for optimum conditions to be achieved, where as the use of pure iron requires more inhibitors to coat metals to optimum conditions. In this study, to coat the entire surface of the metal apparently requires a concentration of inhibitor of 4 g/L to protect the metal under optimum conditions. The more iron content, the higher concentration of corrosion inhibitors needed. The corrosion media that was mixed with inhibitor can decrease corrosion rate due to the adsorption of inhibitors on the surface of the iron to prevent the interaction between iron and corrosion media, in this case is HCl (19).

The ability of pectin inhibitors to reduce the rate of corrosion is due to the presence of carboxyl groups having negatively charged oxygen species in each unit of pectin macromolecules. The negatively charged atom will be adsorbed on the metal surface by performing an electrostatic interaction with the electropositive metal atom present on the iron surface. In addition, the electropositive groups of iron can act as Lewis acid, and therefore can form a coordination bond with oxygen from the pectin macromolecule which acts as a Lewis base.

4 Conclusion

The resulting inquiry-based worksheet on the utilization pectin from lemon peel waste as a corrosion inhibitor displays the inquiry stages, i.e. stages of formulating the problem, making hypotheses, designing experiments, conducting experiments, analyzing data, and making conclusions. Validation test results by three experts concludes that the worksheet is valid, with the average value of rvalue obtained is 0.78 and the average percentage of feasibility is 79.16%. Thus, worksheet made in this research is feasible to be applied in Basic Chemistry course. Pectin from lemon peel is effective to be used as corrosion inhibitor of iron with efficiency at optimum condition that is equal to 78%, at optimum concentration 4g/L with 2 hours immersion time.

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