The Characterization of Co-Ni-Fe Coating in Different pH Environment

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*Abstract***:** *This study aims to produce a Co-Ni-Fe alloy coating on a mild steel hollow cylinder shape by electrodeposition process and analyze the effect of different pH on the corrosion performance of the alloy coating. During the electrodeposition process, the temperature and time are set to 50°C and 30 minutes, respectively. Different pH value consisting of pH 2.1 (strong acid), 5.2 (weak acid), 6.9 (neutral), 8.4 (weak alkali) and 12.3 (strong alkali) are used. From the results obtained it was found that the deposited Co-Ni-Fe alloy has excellent corrosion performance. Corrosion rate obtained for strong acid, weak acid, neutral, weak alkali and strong alkali are 5.88 mmpy, 2.95 mmpy, 1.17 mmpy, 2.16 mmpy and 5.72 mmpy, respectively. Hence this studied showed that Co-Ni-Fe coating alloy could be used as an alternative to prevent against corrosion due to its high corrosion resistance as well as high microhardness.*

Keywords **:** *Co-Ni-Fe, corrosion, electrodeposition, pH value.*

I. INTRODUCTION

Electrodeposition is widely used in the industrial process which include electrocoating, cathodic electrodeposition, anodic electrodeposition and electrophoretic coating. It is one of the most used process in various industries such as in electronics, automotive, aeronautics and piping [1]. Meanwhile, corrosion is known as a metal deterioration that results from the chemical reaction between a metal and its surrounding environment. The most common types of corrosion for unalloyed or alloyed steels are uniform surface corrosion, shallow pit corrosion, pitting corrosion, crevice corrosion, contact corrosion and intergranular corrosion [2], [3].

Corrosion takes place when the metal is oxidized to form metal ions and free electrons. The free electrons will reduce the oxygen which means hydroxyl ions will form and produce cathodic reaction. Anodic reaction will produce ferrous ions and react with hydroxyl ions. From the reaction, ferrous hydroxide is form which then is converted into a hydrated oxide that is known as 'rust'. Based on previous study [4], the effect of pH on the magneto impedance properties of electrodeposited Co-Ni-Fe microtubes was studied. The electrodeposition process was done on the surface of copper wires to produce the cobalt, nickel and iron magnetic samples. The properties of Co-Ni-Fe microtubes were systematically investigated using pH between 2 and 3.5. It was reported that Co-Ni-Fe alloy deposited at higher pH value contained more Ni element than those deposited at lower pH value. This showes that pH affected the magnetic performance of Co-Ni-Fe microtubes. In another study, the effects of pH variation on the corrosion of mild steel in bore-hole water using 1M sodium hydroxide solution, NaOH was reported [5]. The pH values used were only in the alkali range starting from pH 7.2, 8.2, 9.2, 10.2 and 11.2. The mild steel plate was degreased in ethanol, and etched in 5% concentrated hydrochloric acid (HCl) for 30 seconds. The authors found that the sample in bore-hole water with pH value of 11.2 had the least corrosion while the one in bore-hole water of pH 7.2 had the highest corrosion rate. The main issue that is normally faced when using mild steel in any application is its low resistance against corrosion. The cost needed to replace or repair the corroded parts are expensive and thus it is crucial to find a way to prevent the mild steel from corroding. Although there are many techniques available, most of the techniques are not suitable because of the effects and risk that might be harmful to humans and the environment. In this study, Co-Ni-Fe alloy coating will be deposited on a hollow mild steel cylinder and its corrosion behavior in different pH environment will be examined.

II. METHODOLOGY

A. Preparation of Material

The sample used was a mild steel hollow cylinder shape and was cut with a dimension of 35 mm length, 24 mm outer radius and 20 mm inner radius. Silicon carbide paper with 2000 grit was used to remove the black paint covered on the mild steel surface to ease the coating process. The material was cleaned with an ultrasonic clean machine (Delta Ultrasonic Cleaner DC300H) with temperature of 40°C for 4 minutes. All samples were then kept in a sealed container to avoid oxidation.

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B. Preparation of Bath

The bath is a mixture of cobalt sulphate, nickel sulphate, iron(II) sulphate, boric acid, ascorbic acid and saccharine. Saccharine and boric acid were used as a pH buffer and grain refinement agent [6]. The composition for each chemical substance is shown in Table-I. The heating and stirring process was conducted at the same time by using a hotplate magnetic stirrer. The temperature was maintained at 50°C until the mixture dissolves completely.

Table-I: Composition of electrolyte

Chemical Substances	Concentration		
Volume of distilled water	1L	2L	3L
Cobalt Sulphate	14.06 g	28.12 g	42.18 g
Nickel Sulphate	35.04 g	70.08 g	105.12 g
Iron (II) Sulphate	5.56 g	11.12 g	16.68 g
Boric Acid	16.48 g	32.96 g	49.44 g
Ascorbic Acid	11.74 g	23.48 g	35.22 g
Saccharine	1.36 g	2.72 g	4.08 g

C. Electrodeposition Process

The sample was connected to the negative terminal known as the cathode. The steel plate was connected to the positive terminal, anode. The process is done on the LABMART Hotplate Stirrer in order to maintain operating temperature which was 50°C and 2.0 A current [7]. The time taken for electrodeposition to take place was 30 minutes and it was kept constant for all samples. After the electrodeposition process was completed, the sample was rinsed with distilled water, dried and stored in a sealed container to avoid oxidation.

D. Characterization Technique

Scanning Electron Microscope was used to study the surface topography. The material composition was also obtained using Energy Dispersive X-ray (EDX). Vickers microhardness test was carried out with load of 1kg for 10 seconds on the selected area of the sample surface. The reading was taken 5 times at different spots of the sample in order to ensure the accuracy of measurement [8]. The average value of microhardness was and plotted in a graph.

E. Corrosion Test

The method used to obtain the corrosion rate of Co-Ni-Fe alloy was potentiodynamic polarization technique. The corrosion test consists of counter electrode, reference electrode, and working electrode. The solution was prepared according to different pH value which ranges from strong acid until strong alkali as shown in Table-II. Before potentiodynamic polarization (PDP) is done, open circuit potential (OCP) was first carried out. The OCP required about one hour. The Tafel extrapolation was plotted to obtain the corrosion rate based on the result of the PDP test [9].

Table-II: Types of solution with different pH values

pH values	Solution
2.1	Hydrochloric acid, HCl
5.2	Hydrofluoric acid, HF
6.9	Distilled water
8.4	Salt water
12.3	Sodium hydroxide, NaOH

III. RESULTS AND DISCUSSION

A. Microhardness

Table 3 shows the microhardness for samples before and after the corrosion test in different pH environment. It can be seen that the value of microhardness of Co-Ni-Fe coating alloy after corrosion test for all pH environments is much lower as compared to the microhardness before corrosion test. The value increases in neutral condition and then decreases again in pH 8.4 and 12.3. This is due to the corrosion rate during PDP where corrosion rate is high in acidic and high alkaline solutions.

Table-III: Microhardness of mild steel in different pH condition

Condition	pH value	Hardness, HV
Before corrosion	-	338.96
After corrosion	2.1	242.34
	5.2	232.06
	6.9	289.38
	8.4	265.5
	12.3	250.94

The cause of the decrease in microhardness is also due to the existence of grain boundaries. Based on previous study, grain boundaries increases the size of the grains that causes reduction in hardness in surface materials [10]. After the PDP test, the pH 6.9 shows the highest value of hardness which is 289.38 HV while pH 5.2 shows the lowest value of hardness when the value is 232.06 HV. From the result obtained it can be seen that corrosion causes the microhardness value to reduce. It has been reported that the presence of higher nickel weight percentage in the alloy improves its corrosion resistance [11]. As pH value increases from 7 until 14, the amount of microhardness of Co-Ni-Fe alloy decreases. Furthermore, the decrease of hardness for coatings with higher Fe content in pH 12.3 is due to the solid solution hardening.

B. Morphology and Microstructure

Scanning Electron Microscope (SEM) was used to study the morphology and microstructural characterization of Co-Ni-Fe alloy coating before and after corrosion.

Fig. 1 shows the microstructure of Co-Ni-Fe coated sample before corrosion. It can be seen that it has uniform surface coating without any pores visible. Thus the hardness and corrosion resistance of Co-Ni-Fe alloy coating was high. Fig. 2, 3, 4, 5 and 6 show the microstructure of the coated samples after undergoing corrosion test in solutions with pH 2.1, pH 5.2, pH 6.9, pH 8.4 and pH 12.3, respectively. After the PDP test were conducted the sample showed the signs of corrosion on the coating surface and presence of pores at certain spots could be seen where it is believed that this had allowed the substrate to be exposed to surrounding which caused corrosion to happen and reduce its mechanical properties [12].
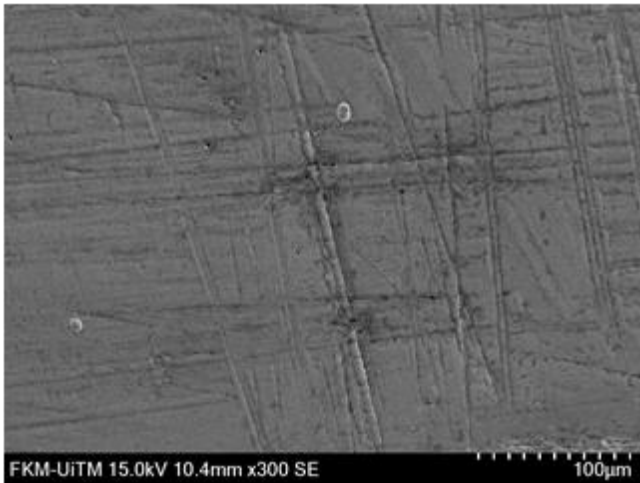
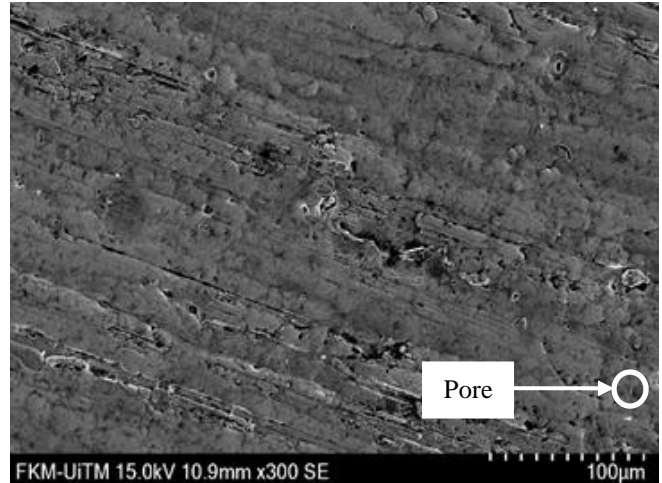**Fig. 1: Surface of Co-Ni-Fe coating before PDP was conducted**

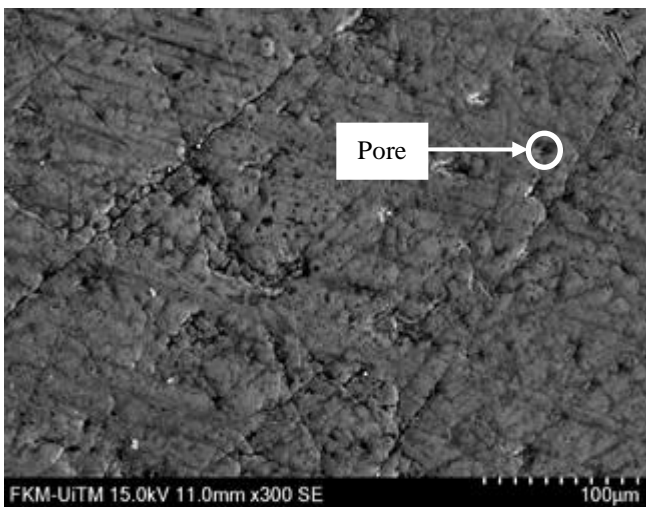**Fig. 4: Surface of Co-Ni-Fe coating after PDP in pH 6.9 solution**

Fig. 2: Surface of Co-Ni-Fe coating after PDP in pH 2.1 solution
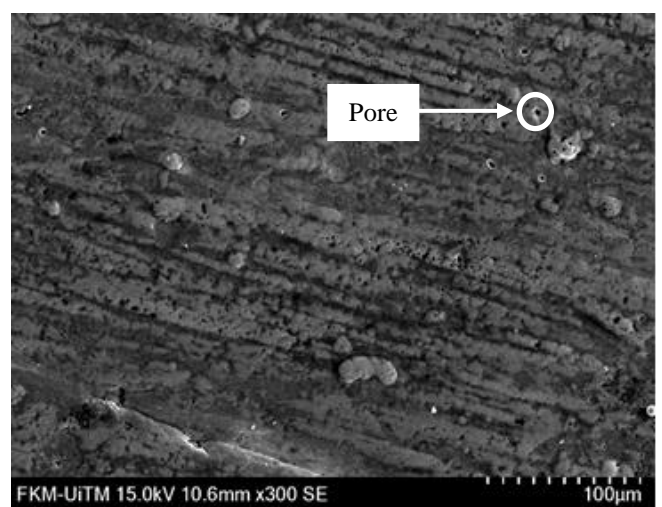

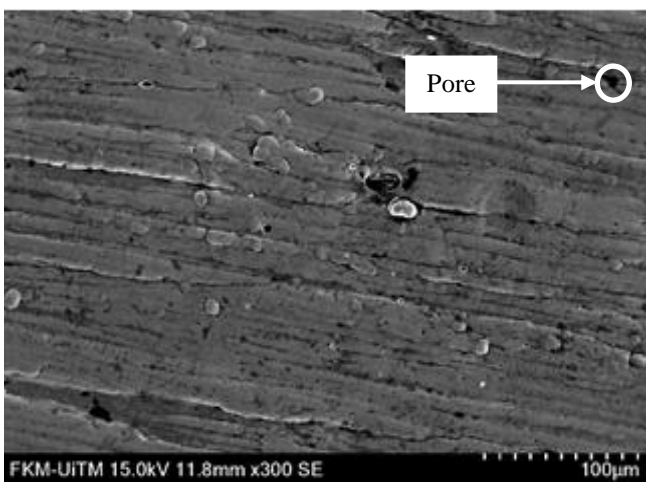**Fig. 5: Surface of Co-Ni-Fe coating after PDP in pH 8.4 solution**

Fig. 3: Surface of Co-Ni-Fe coating after PDP in pH 5.2 solution
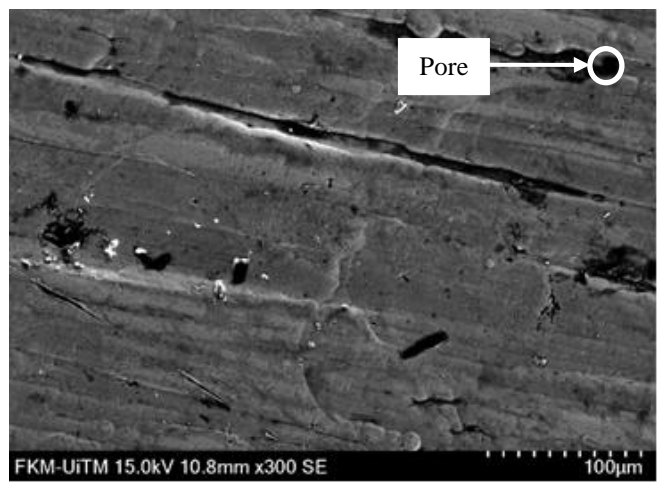

Fig. 6: Surface of Co-Ni-Fe coating after PDP in pH 12.3 solution.

Energy Dispersive X-ray (EDX)

Energy Dispersive X-ray is used to observe the composition of the Co-Ni-Fe alloy coating on the mild steel cylinder.

The difference in EDX peaks is influenced by the morphological difference of surface coating for each material. Table-IV shows the composition of material on the surface for all samples in both conditions.

The presence of carbon (C) in all samples is due to the usage of carbon tape during preparation for EDX testing [2]. Meanwhile, presence of oxygen (O) is due to the oxidation process that occurs and causes the corrosion to happen. All samples also showed the presence of Cobalt (Co), Nickel (Ni) and Iron (Fe). From previous study [13], the researchers reported that the usage of saccharine as the grain refinement agent contributes to the presence of sulphur. However in this study, there was no presence of sulphur (S) in the Co-Ni-Fe alloy coating.

Fig. 7 shows the element composition against pH condition. After corrosion, the highest weight percentage for nickel was located at pH 6.9 (neutral). For cobalt element, the highest value of the element is located at pH 12.3 which is 46.7%. Meanwhile, the highest amount of iron element is located at pH 12.3 which is 20.2%. The iron element plays a major role in increasing the hardness of material while nickel will increase the corrosion performance of materials.

Table-IV: The composition of elements in Co-Ni-Fe alloy in difference pH environment

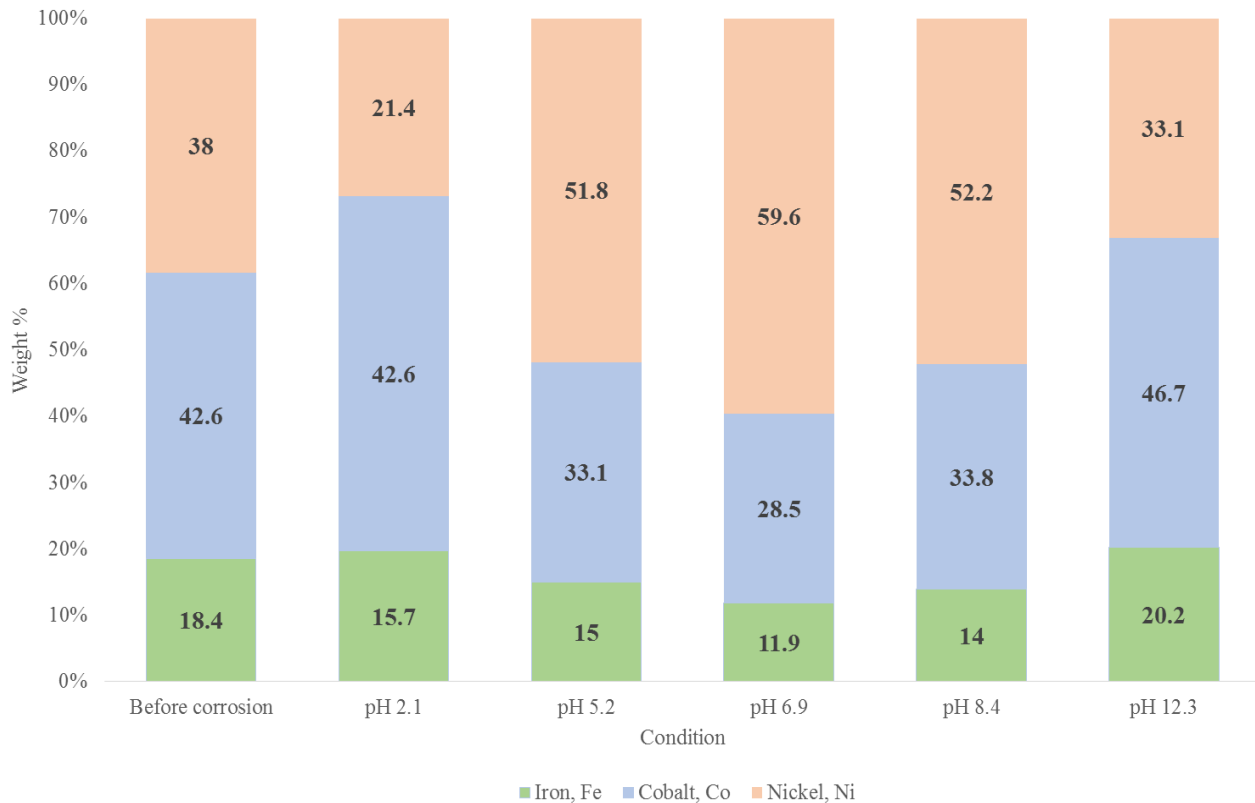| Condition	Before corrosion	After corrosion (pH 2.1)	After corrosion (pH 5.2)	After corrosion (pH 6.9)	After corrosion (pH 8.4)	After corrosion (pH 12.3)
Elements	Weight%	Weight%	Weight%	Weight%	Weight%	Weight%
Fe	18.4	15.7	15.0	11.9	14.0	20.2
Co	42.6	42.6	33.1	28.5	33.8	46.7
Ni	38.0	21.4	51.8	59.6	52.2	33.1

Fig. 7: Weight of element composition against condition

C. Potentiodynamic Polarization Test

The potentiodynamic polarization was conducted in order to obtain the corrosion rateof the alloy coating in different pH environment. Open circuit potential was conducted prior to the potentiodynamic polarization test and is shown in Fig. 8. OCP is done in order to conduct and evaluate the stability and make the sample's potential stable before performing potentiodynamic polarization. Fig. 9 shows the PDP result obtained for Co-Ni-Fe alloy coatings in pH 5.2.Tafel plot was then used to measure the corrosion rate of all samples and is shown in Fig. 10.
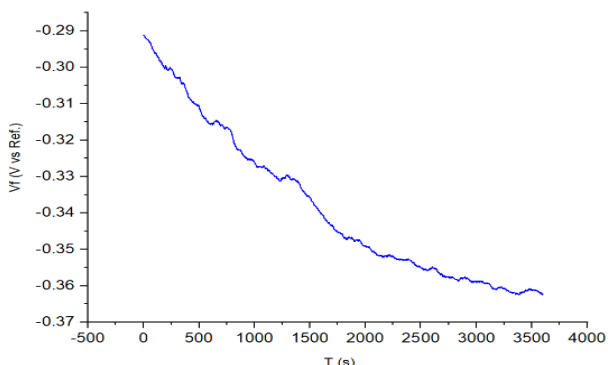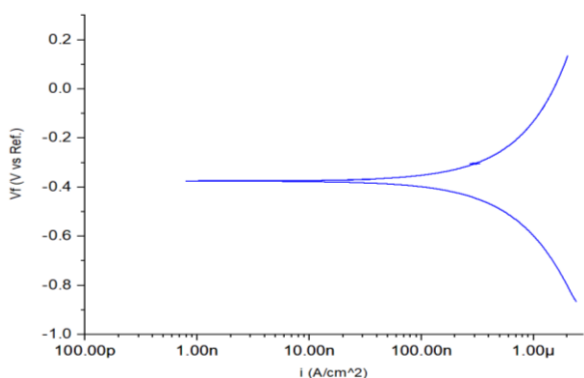

Fig. 8: Open circuit potential graph

Fig. 9: Potentiodynamic polarization test result for Co-Ni-Fe alloy coating in pH 5.2
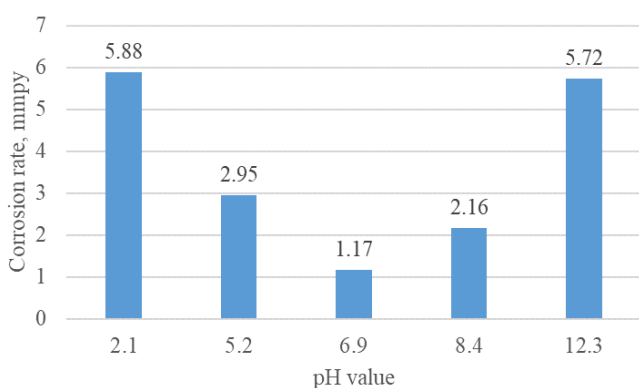

Fig. 10: The corrosion rate in different pH environment

Table-V tabulates the corrosion rate of samples in different pH conditions. For pH 2.1 which is the strong acid indicates the highest value of corrosion rate with value of 5.88 mmpy while pH 6.9 which is neutral showed the lowest rate of corrosion value of 1.17 mmpy. Low corrosion rate is due to the compactness of microstructure and smallest grain size

[14]. Strong acid increases the rate of corrosion of Co-Ni-Fe alloy coatings as compared to the low pH environment where similar results were also reported by Zainab et al [15].

Table-V: Corrosion rates in different pH value

pH value	Corrosion rate (mmpy)	I_{corr} (A/cm^2)	E_{corr} (mV)
2.1	5.88	0.00617	-568
5.2	2.95	0.00315	-375
6.9	1.17	0.06195	-353
8.4	2.16	0.11452	-842
12.3	5.72	0.30241	-1220

IV. CONCLUSION

This study has successfully Co-Ni-Fe coating on the mild steel cylinder through the electrodeposition process. The different pH environment significantly effects the corrosion rate of the Co-Ni-Fe alloy coating. Strong acid gives the highest corrosion rate while neutral condition produces the lowest corrosion rate. Corrosion rate obtained for strong acid, weak acid, neutral, weak alkali and strong alkali are 5.88 mmpy, 2.95 mmpy, 1.17 mmpy, 2.16 mmpy and 5.72 mmpy, respectively.

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