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# FTIR Analysis on Phase Transformation of Rust In The Presence of Gambir

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## Abstract

The performance of gambir reacted with rust by means of FTIR was evaluated. The effects of gambir concentration, contact time and the addition of phosphoric acid in phase transformation of rust were studied. The FTIR spectra showed that the conversion of lepidocrocite ( $\gamma$ -FeOOH) was the fastest, followed by magnetite ( $\text{Fe}_3\text{O}_4$ ) and goethite ( $\alpha$ -FeOOH) was the slowest. 10% gambir with 3 h contact time was found to be the optimum condition for the phase transformation. Addition of phosphoric acid gave a better performance in phase transformation of rust.

**Keywords:** FTIR; Gambir; Phase Transformation; Rust

## 1. Introduction

Rust products are complex materials that change continuously as the complex develops through the precipitation, evolution and transformation of chemical species in the iron-oxygen-water system. Under ambient condition, lepidocrocite ( $\gamma$ -FeOOH), goethite ( $\alpha$ -FeOOH) and magnetite ( $\text{Fe}_3\text{O}_4$ ) are the main constituents of rust and the specific composition each depends on the pH, temperature and the presence or absence of atmospheric pollutants [1]. Rust converter is one of the efforts in controlling corrosion, preventing corrosion from further occurring. It can be applied on the steel surface, serves as barrier to protect the steel as well as a base for paint.

Recently, few studies had been conducted in order to evaluate commercial rust converter. Several factors affect the mechanisms of the conversion including the components of the converter, pH, and reaction time of the converter with the rust as well as the method of application [2]. Besides rust

converters that are based on phosphoric acid and chromate, researchers are now more interested into the study of natural inhibitors as more ecofriendly and biodegradable rust converters such as gallic acid and tannic acid [3],[4]. The naturally occurring polyphenols mostly in plants can react with iron ions lead to more stable, insoluble iron complexes so that no further reaction can occur between rust and the corrosive agents [5].

Gambir (*Uncaria gambir*) consists of catechin as the most abundant polyphenolic constituent [6]. It was found that due to its high catechin contents, gambir possesses high antioxidant capacities [7], [8]. Other researchers found other applications of gambir plant including inhibitors in preventing corrosion of mild steel in acidic solution [9] as adsorbent of selected dye adsorption [10] and one of the components in mouth antiseptic [11]. Catechin polyphenols have been demonstrated to act directly as radical scavengers of oxygen and nitrogen species besides having well established metal chelating properties [12].

## 2. Material and Methods

### 2.1 Preparation of gambir extract

The gambir cubes were purchased from a local market in Penang, Malaysia. The cubes were ground into powder form with the mesh size of 250  $\mu\text{m}$ . Approximately 5 g of defatted gambir was mixed with ethyl acetate (1:10 m/v) incubated for 3 h at 200 rpm under maceration and at room temperature. Then, the extracts were concentrated by rotary evaporation under reduced pressure (Heidolph Instruments, Germany) and the resulting solution was dried and kept in closed container for further use.

### 2.2 Fourier transform infrared spectroscopy (FTIR) analysis

All samples were prepared by mixing 5 mg of sample with 100 mg of potassium bromide (KBr, Merck) and pressed to form a thin transparent pellet. The investigation was carried out within the wavenumber ranging from 4000  $\text{cm}^{-1}$  to 400  $\text{cm}^{-1}$  by using Perkin-Elmer System 2000 Spectroscopy (Sigma Aldrich, USA). All the rust standards (lepidocrocite, goethite, and magnetite) were commercially purchased (Sigma Adlrch).

### 2.3 Preparation of rust powder

Mild steel plates with chemical compositions (wt %) of 0.08 C, 0.01 Si, 1.26 Mn, 0.02 P and remaining Fe were used. The plates (6 cm x 4 cm x 1 mm) were cleaned with sand paper, rinsed with distilled water and isopropyl alcohol and allowed to dry at room temperature. The plates were then exposed in salt spray chamber (Standard Salt Spray Tester ST-BZ-7, Elite Test Instrument Co. Ltd, Taiwan) containing of 5% NaCl (Merck) for 24 h according to ASTM B117 standard. After that, the plates were rinsed with distilled water and were dried in an oven at 40°C for 24 h. The rust that was produced on the steel was removed with a scrapper, ground into powder form and kept in closed container for further studies.

### 2.4 The effect of concentration of gambir solution on rust treatment

A 50 mg of rust powder was mixed with 10 mL of gambir extract in methanol solution. The concentrations of gambir extract solution were 1%, 5%, 7%, 10%, 15% and 20% (w/v). The solutions were at pH 5, which is the natural

pH for gambir solution. The mixtures were mixed and were allowed to react at room temperature for 1 h. After that, the mixtures were filtered and the filtered residues were dried in an oven at 40°C for 1 h. The dried samples were collected and analyzed by FTIR.

### 2.5 The effect of reaction time of gambir solution on rust treatment

A set of 5 solutions of 10 mL of gambir extract in methanol was prepared. The concentration (10% (w/v)) and pH (pH 5) of the solution were kept constant. 50 mg of rust powder was mixed into each solution and allowed to react at room temperature. The reaction times studied were at 1 h, 2 h, 3 h, 6 h and 12 h. The samples were then filtered, dried in oven and analyzed with FTIR.

### 2.6 The effect of addition of phosphoric acid on rust treatment

A set of 10 mL solution of gambir extract in methanol mixed with phosphoric acid (1:1, v/v) was prepared. The concentration of gambir solutions were kept constant at 10% (w/v) while the concentration phosphoric acid ( $\text{H}_3\text{PO}_4$ , R&M Chemicals) solutions were varied from 3% to 15% (w/v). 50 mg of rust powder was mixed into each solution and allowed to react at room temperature for 3 h. The samples were then filtered, dried in oven and analyzed with FTIR.

### 2.7 Surface analysis

Surface analyses were performed using scanning electron microscopes (SEM, Leo Supra 50VP Field) at 3000X magnification equipped with an energy-dispersive X-ray spectroscopy (EDX, Oxford INCA 400) to observe the physical changes on the samples.

## 3. Result and Discussion

### 3.1 Characterization of rust by FTIR

Fig. 1 depicts the FTIR spectra of rust standards; lepidocrocite ( $\gamma\text{-FeOOH}$ ), goethite ( $\alpha\text{-FeOOH}$ ) and magnetite ( $\text{Fe}_3\text{O}_4$ ). Adsorption band around 1650  $\text{cm}^{-1}$  indicated the adsorbed  $\text{H}_2\text{O}$  in almost all samples [1].

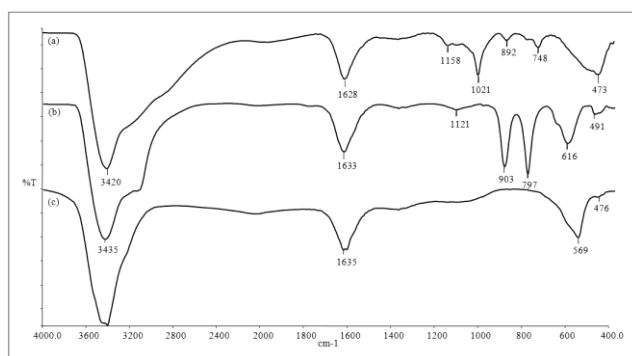


Fig. 1 FTIR spectra of rust standards; (a) lepidocrocite, (b) goethite and (c) magnetite.

Lepidocrocite has bands in its FTIR spectrum at 1158  $\text{cm}^{-1}$  (medium), 1021  $\text{cm}^{-1}$  (strong), 892  $\text{cm}^{-1}$  (medium), 748  $\text{cm}^{-1}$  (medium) and 473  $\text{cm}^{-1}$  (strong). Thus, 1021  $\text{cm}^{-1}$  is the strongest and could be considered as lepidocrocite's major band. According to [1], well crystalline lepidocrocite has bands in its spectrum at 1150, 1020 and 750  $\text{cm}^{-1}$  for O-H bending.

The observed goethite FTIR spectrum has peaks located at 1121  $\text{cm}^{-1}$  (weak), 903  $\text{cm}^{-1}$  (strong), 797  $\text{cm}^{-1}$  (strong), 616  $\text{cm}^{-1}$  (medium) and 491  $\text{cm}^{-1}$  (weak). The peaks located at 903  $\text{cm}^{-1}$  and 797  $\text{cm}^{-1}$  are very strong and sharp. These are considered as major characteristic peaks for goethite. In its most stable state, goethite has adsorption peaks at 1644, 1386, 890, 769 and 650  $\text{cm}^{-1}$  [1].

Magnetite's FTIR spectrum contained a strong peak at 569  $\text{cm}^{-1}$  and a small shoulder peak at 475  $\text{cm}^{-1}$  (weak). The observed peaks matched with the study found by [1] and [13], that magnetite contained two major characteristic peaks at 568 and 473  $\text{cm}^{-1}$ . Ref. [4] and [14] reported that these bands can be assigned to Fe-O stretch.

Fig. 2 shows the spectrum of rust generated by salt spray. It was observed that the major products of steel rust were  $\gamma\text{-FeOOH}$ ,  $\alpha\text{-FeOOH}$  and  $\text{Fe}_3\text{O}_4$ . It is in agreement with the study by [13] and [15] who reported that those products were the major constituents of any rust system formed on steel surfaces under normal ambient conditions. Ref. [4] also reported that the rust components depend on the method of preparation.



Fig. 2 FTIR spectrum of rust generated by salt spray (L = lepidocrocite, G = goethite, M = magnetite).

Peak at 1620  $\text{cm}^{-1}$  belongs to the adsorbed  $\text{H}_2\text{O}$ . The peaks at 1021, 745 and 470  $\text{cm}^{-1}$  belong to lepidocrocite. These peaks showed the same intensity absorption with the standard, but with slightly different frequencies, that may be due to purity of the sample and different crystallinity of  $\gamma\text{-FeOOH}$ . Two major characteristic peaks of goethite were located at 890 and 796  $\text{cm}^{-1}$ , both with medium intensity. The presence of magnetite was identified at 547  $\text{cm}^{-1}$  with strong intensity.

### 3.1 The effect of concentration of gambir solution

The rate of transformation (% RT) of rust was calculated according to the following formula:

$$\text{Rate of transformation, \%} = \frac{\ell_0 - \ell_1}{\ell_0} \times 100 \quad (1.0)$$

Where  $\ell_0$  is the intensity of the control peak (rust before treatment) and  $\ell_1$  is the intensity of the sample peak (rust after treatment).

The FTIR spectra of rust powder treated with gambir extract of varied concentration (1% to 20%) are shown in Fig. 3. It was observed that the intensity of each of the rust constituent was reduced after 1 h immersion in gambir solution at pH 5. The intensity of lepidocrocite major peak located at 1021  $\text{cm}^{-1}$  reduced significantly up to 77.6% RT for 20% gambir. The other peaks of lepidocrocite at 745 and 470  $\text{cm}^{-1}$  were 80.0% transformed and shifted starting from 10% gambir solution. The peaks of goethite at 890 and 797  $\text{cm}^{-1}$  were slightly reduced and seem to be transforming slower than lepidocrocite peaks. The RT was increased up to 67.5% and 69.2% for peaks 870  $\text{cm}^{-1}$  and 797  $\text{cm}^{-1}$ , respectively. The peak of magnetite had become broader and the frequency was shifted from 547  $\text{cm}^{-1}$  to 616  $\text{cm}^{-1}$  in 15% and 20% gambir with RT was up to 50.0%. Based on the spectra and RT, 10% gambir concentration was selected as optimum concentration for rust powder treatment.

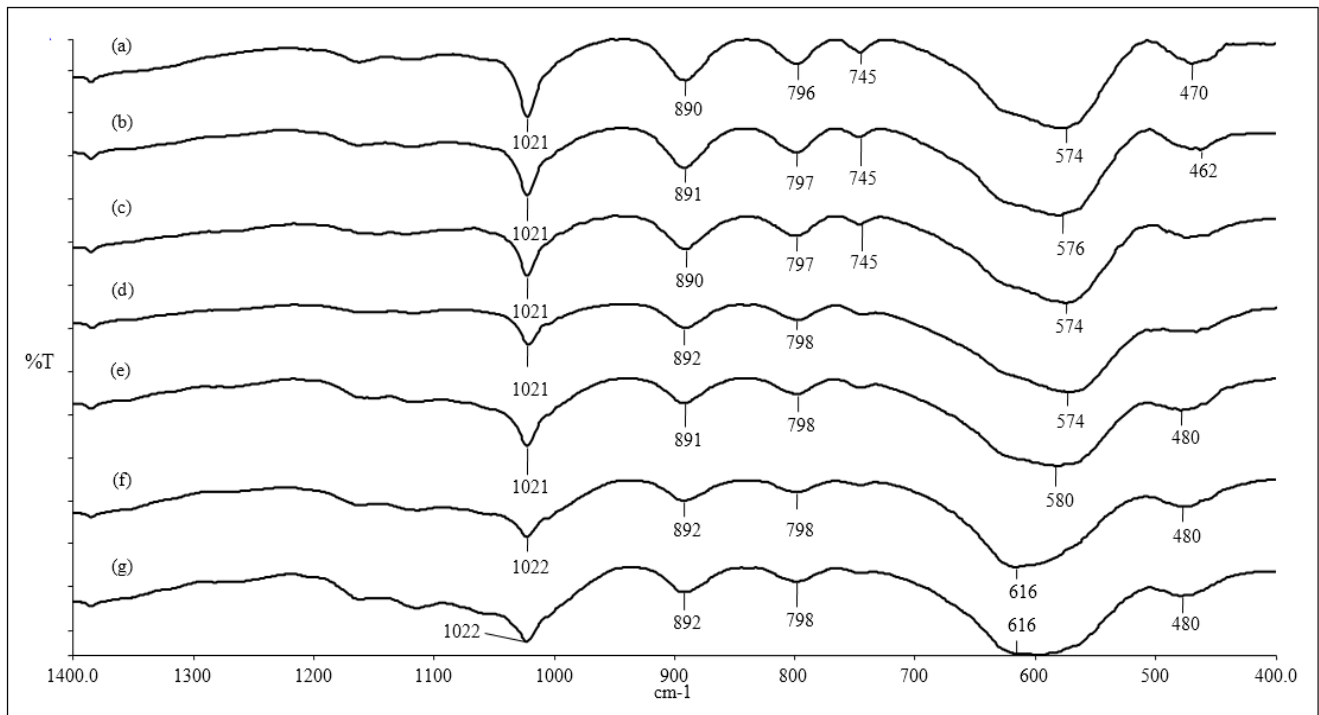


Fig. 3 FTIR spectra of (a) rust before treatment and rust after 1 h treatment with (b) 1%, (c) 5%, (d) 7%, (e) 10%, (f) 15% and (g) 20% gambir.

### 3.2 The effect of reaction time

The time selected in this study was 1 h, 2 h, 3 h, 6 h and 12 h. Fig. 4 depicts the spectra of rust after treated with 10% gambir at pH 5 for various time immersions. The spectra show obvious changes in the intensity of each rust constituent as the time of immersion increased.

It was observed that beginning 3 h of contact time with the solution, the peak of  $1021\text{ cm}^{-1}$  of lepidocrocite disappeared and became broader after 6 h immersion. New peaks at  $1156$  and  $1116\text{ cm}^{-1}$  are referred to the formation of ferric tannate [1], [16]. The peak at  $745\text{ cm}^{-1}$  was completely removed after 3 h while the peak at  $470\text{ cm}^{-1}$  remained the same throughout the time, which shifted after 1 h immersion. Both goethite peaks at  $890$  and  $796\text{ cm}^{-1}$  show a comparable rate of transformation. The peak at  $890\text{ cm}^{-1}$  start to transforms with 64.0% to 84.6% RT while the peak at  $796\text{ cm}^{-1}$  was transformed from 61.5% to 84.6% RT after 12 h treatment. The adsorption band of magnetite located at  $574\text{ cm}^{-1}$  was transformed with 50.0% RT up to 65.4% after 3 h immersion. It was observed that after 6 h of contact time, the magnetite peak was shifted from  $574\text{ cm}^{-1}$  to  $618\text{ cm}^{-1}$ .

It can be concluded that after 3 h of reaction time with 10% gambir, the rust can substantially form an iron complex [17]. The gambir reaction with rust was faster with lepidocrocite, slower with magnetite and slowest with goethite. This was in agreement with [1], [4] and [13]. Ref. [16] stated that in the presence of tannins, lepidocrocite undergoes a reductive dissolution under formation of ferrous complex, which oxidized to precipitate insoluble ferric tannate.

Ref. [18] proposed three ways of tannin reaction with iron: (1) the tannins can complex with  $\text{Fe}^{2+}$  ions to form ferrous-tannates which can easily oxidized into ferric-tannates when in contact with oxygen, (2) the tannin can react directly with the  $\text{Fe}^{3+}$  ions to form ferric-tannates, and (3) tannin can reduce  $\text{Fe}^{3+}$  oxides into  $\text{Fe}^{2+}$  ions, then the tannin will complex with  $\text{Fe}^{2+}$  ions, thus oxidized into ferric-tannates in the presence of oxygen. The  $\text{Fe}^{3+}$  in equilibrium with  $\text{FeOOH}$  are reduced to  $\text{Fe}^{2+}$  ions when in contact with iron metal in the pores of the rust layer and these  $\text{Fe}^{2+}$  compounds can be easily re-oxidised into  $\text{Fe}^{3+}$  oxides.

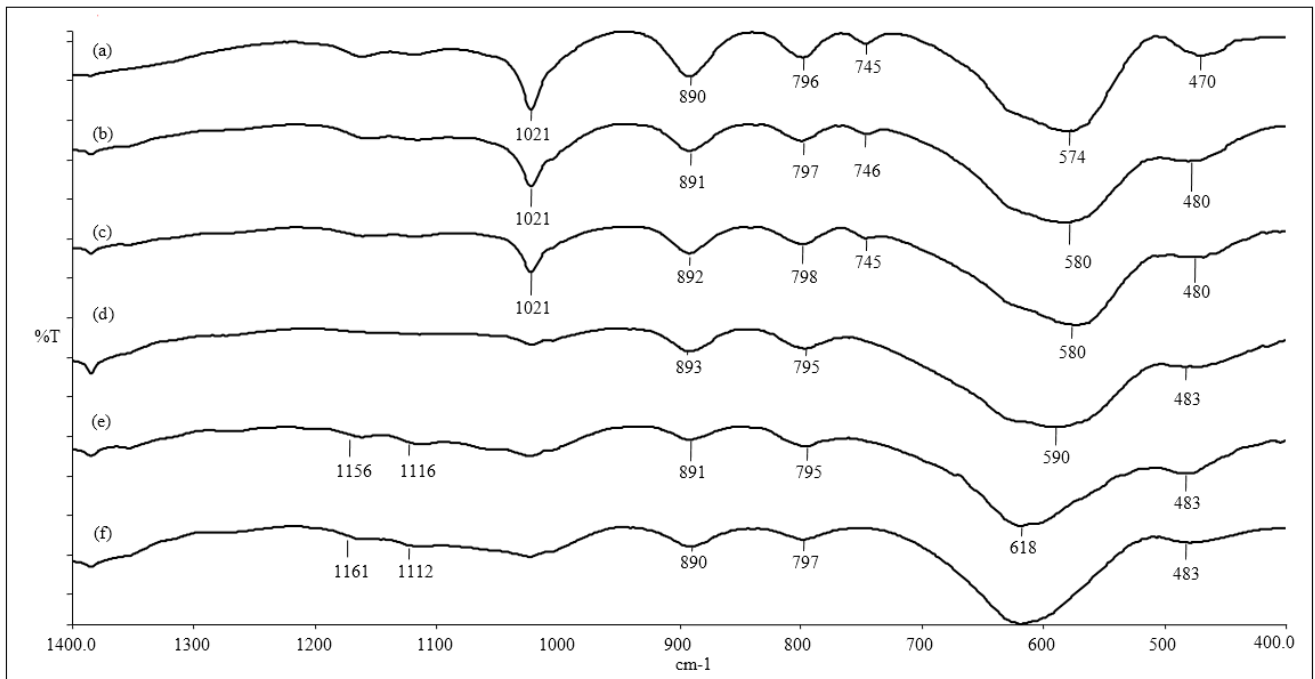


Fig. 4 FTIR spectra of (a) rust before treatment and rust after treatment with 10% gambir for (b) 1 h, (c) 2 h, (d) 3 h, (e) 6 h and (f) 12 h contact time

### 3.3 The effect of addition of phosphoric acid

Phosphoric acid,  $H_3PO_4$ , was selected in combination with gambir because it is known as one of the oldest types of rust converters. Fig. 5 shows and the spectra of rust when different concentrations of  $H_3PO_4$  (3% to 15%, v/v) were added into the treatment solution. It was observed from the spectra, as compared to rust treated in 10% gambir alone, the addition of  $H_3PO_4$  reduced the intensity of rust significantly better after 3 h immersion.

Lepidocrocite peak at  $1021\text{cm}^{-1}$  become broader in addition of  $H_3PO_4$ . This was due to the formation ferric phosphate [1], [19]. The adsorption band of lepidocrocite at  $745$

$\text{cm}^{-1}$  was completely removed. The other peak at  $470\text{cm}^{-1}$  was slightly shifted to  $480\text{cm}^{-1}$ . The same pattern was observed with magnetite adsorption band at  $574\text{cm}^{-1}$  which shifted to  $617\text{cm}^{-1}$ . However, there were not much changes observed with goethite peaks. Both goethite peaks had more than 80.0% RT. The reduction of rust intensity basically showed the same behaviour from additional of 3% to 15%  $H_3PO_4$ .

Therefore, it can be concluded that the addition of  $H_3PO_4$  into the solution treatment can increase the degree of transformation of rust. Other authors also concluded that the addition of  $H_3PO_4$  in conjunction with tannin can perform better conversion [1], [13], [20].

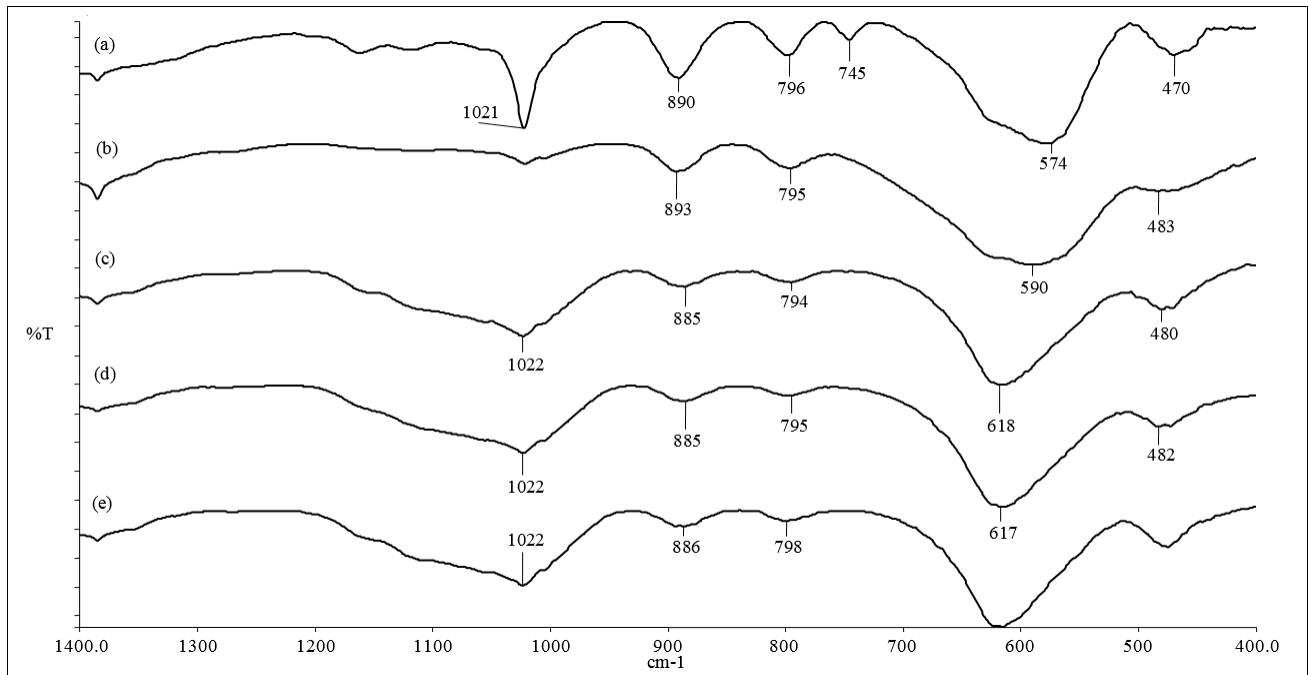


Fig. 5 FTIR spectra of (a) rust before treatment and rust after 3 h treatment with (b) 10% gambir, (c) 10% gambir + 3%  $\text{H}_3\text{PO}_4$ , (d) 10% gambir + 5%  $\text{H}_3\text{PO}_4$  and (e) 10% gambir + 15%  $\text{H}_3\text{PO}_4$

### 3.4 Comparison of rust powder treatment

Fig. 6 shows the comparison of the rust's spectra treated with 5%  $\text{H}_3\text{PO}_4$ , 10% gambir and mixed solution of 10% gambir + 5%  $\text{H}_3\text{PO}_4$  after 3 h and 24 h reaction time. It was noticed that after 3 h immersion, the treatment of 5%  $\text{H}_3\text{PO}_4$  did not give much effect in reducing the intensity of rust constituents as compared to 10% gambir and mixture of 10% gambir + 5%  $\text{H}_3\text{PO}_4$ . After 24 h treatment, the 5%  $\text{H}_3\text{PO}_4$  infrared spectrum shows the magnetite peak at  $547\text{ cm}^{-1}$  shifted to  $617\text{ cm}^{-1}$  and lepidocrocite peak at  $470\text{ cm}^{-1}$  shifted to  $481\text{ cm}^{-1}$ . No peak seems to disappear. The peak at  $1021\text{ cm}^{-1}$  became broader and there's a new peak appeared due to the formation of ferric phosphate [1], [19]. Other rust peaks ( $890, 796, 745\text{ cm}^{-1}$ ) basically reduced in intensity.

The infrared spectrum of 10% gambir shows the ap-

pearance of peaks at  $1117, 1084$  and  $670\text{ cm}^{-1}$  after 24 h contact time. The other peaks seem to intensify as compared to 3 h contact time. The FTIR spectrum of the mixture of 10% gambir + 5%  $\text{H}_3\text{PO}_4$  showed the appearances of adsorption bands after 24 h treatment at  $1112, 1054, 1004$  and  $670\text{ cm}^{-1}$  due to the ferric tannate and ferric phosphate complexes. Ref. [1] reported that the ferric phosphate complexes caused the broadening of the band between  $1000$  and  $1200\text{ cm}^{-1}$ . The shifted and intensified magnetite peak as well as the visible band at  $670\text{ cm}^{-1}$  is believed to be overlapping with catechin, confirming the formation of ferric tannate.

Therefore, it can be concluded that the treatment of rust with  $\text{H}_3\text{PO}_4$  alone does not significantly transform the rust as supported by the findings of [19]. Gambir mixed with  $\text{H}_3\text{PO}_4$  clearly seem to be a better rust converter.

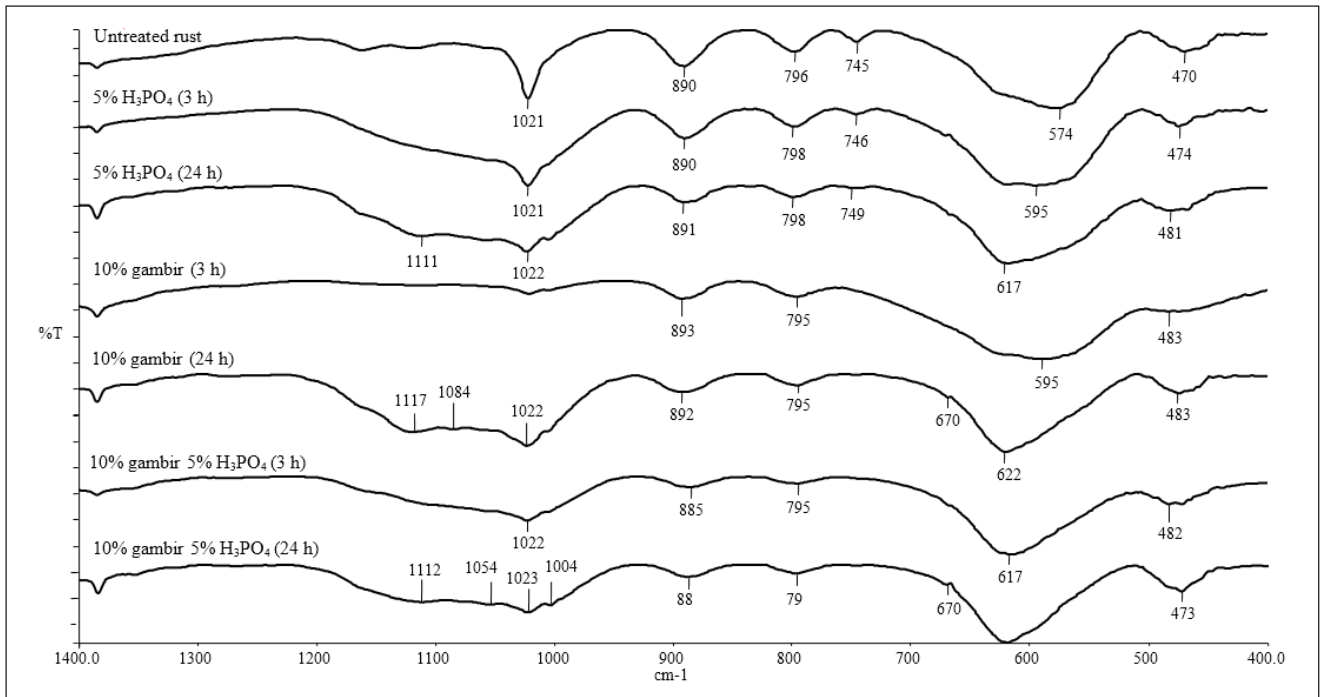


Fig. 6 FTIR spectra of rust before treatment and rust after treatment with 5%  $H_3PO_4$ , 10% gambir and 10% gambir + 5%  $H_3PO_4$  for 3 h and 24 h reaction time.

### 3.5 Surface analysis

Fig. 7 shows the results of SEM with EDX of rust before and after 3 h treatment with rust converter. The morphology of rust before treatment was significantly changed into more homogeneous and compact surface after treated with gambir and mixture of 10% gambir + 5%  $H_3PO_4$ . Ref. [21] reported that the morphologies of rust were cactus/spike shapes and granular shapes indicating the presence of lepidocrocite and goethite. Hence, the application of the converters to the rusted steel surface forms an amorphous compact layer. However, when the rust was treated with 5%  $H_3PO_4$  alone, the surface is in irregular shape and there were still traces of cactus shapes as in the rust before treatment.

The EDX analysis of rust shows traces of the peaks of chloride ions, which are originated from NaCl that was used for generating the rust sample by salt spray chamber. After treatment with rust converter, the carbon and phosphorus peak increased indicating the presence of gambir and phosphate, respectively.

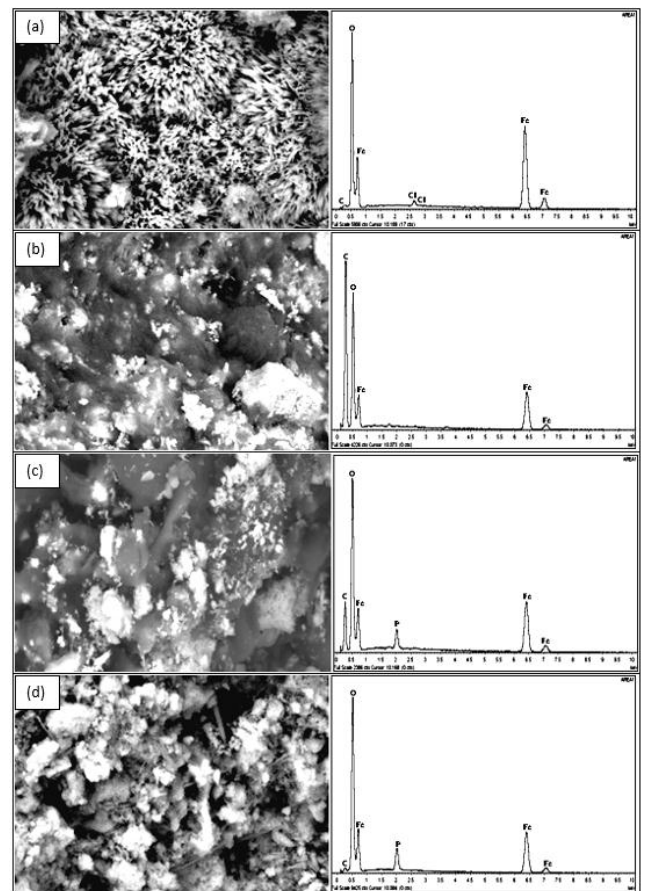


Fig. 7 SEM-EDX analyses of (a) rust before treatment and rust after treatment with (b) 10% gambir, (c) 10% gambir + 5%  $H_3PO_4$  and (d) 5%  $H_3PO_4$



## 4. Conclusion

The treatment of rust powder with gambir extract showed that after 3 h treatment in 10% gambir solution, the rust constituent reduced significantly. There were also new peaks indicating the presence or formation of ferric tannate. The addition of phosphoric acid into the solution gave a better degree of conversion as depicted in the FTIR with a broader peaks and the appearance of new adsorption bands. This also indicated the formation of ferric phosphate besides ferric tannates. The order of transformation of rust from fast to slow: lepidocrocite > magnetite > goethite.  $H_3PO_4$  alone was not convenient in transforming rust after 3 h treatment. Combining gambir and phosphoric acid gave a significant phase transformation of rust. The surface analysis of SEM showed a more compact and homogeneous layer of rust after reacted with gambir and phosphoric acid.

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