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Research paper

Evaluation of Lacquered Tinplate Corrosion in Canned Food through Characterization by using SEM & EDS Technique

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Abstract

Investigation of tinplate corrosion occurs in the interior of Cans containing food products due to electrochemical reactions. This changing the organoleptic properties of food stuff and produced toxicological problems. To investigate corrosion effect, lacquer adhesion failure of tinplate containers was evaluated with Scan Electron Microscope (SEM) along with Energy Dispersive X-ray spectroscopy (EDS) technique. This technique plays a vital role to investigate the reason of corrosion occurs in cans (container) like stress corrosion in the pack of fish and potatoes, pitting corrosion in the pack of light colored fruits and failure due to lacquer defects. Support of analytical methods may be also required to establish the failure.

Keywords: *Tinplate, Lacquer, Scan Electron Microscope, Dispersive X-ray Spectroscopy, Corrosion*

1. Introduction

Tinplate is widely used for packaging and transportation of foods [1, 2] especially for food packing (Crnčević, 1980). One of the most common uses of tinplate is the manufacture of food-cans. The main goal of these cans is to protect foodstuff against chemical and physical risks, until the product is consumed. Tinplate corrosion resistance plays an important role in the shelf-life of the canned product, its appearance and taste. In order to obtain adequate shelf-life of canned product, it is important to select the appropriate tinplate substrate for the food product.
Long term unrefrigerated storage of many vegetables, meat and beverages would be impossible without tinplate cans. The tinplate cans are fabricated form low carbon mild steel sheet with a thin coat of electrodeposited tin [3]. About one third of the world tin production goes to manufacture of tin cans (food containers). More than 2.5 million of tin cans are consumed every year in the United Kingdom. As well as, 25% of worldwide production of steel used to produce beverage tinplate cans [4]. Fabech (1998) recorded that, food producer are used lacquered cans for various category of foods, which including water, beverage, soft drinks, some alcohol drinks (beer and wine), fruit juices, nectars, tomato and tomato products [5]
According to Kamm [6], corrosion evolution in tinplate is controlled by the continuity of the layers of free and alloyed tin (intermetallic), as well as by the behavior of exposed steel as detinning advances. A high porosity of the tin coating in tinplate has been related to high corrosion rates, and may shorten shelf-life [7].

The presence of discontinuities in tin coatings may cause the same effect, by increasing the number of sites for galvanic corrosion due to a higher cathodic area (exposed iron), which promotes corrosion reactions.

Tin cans hold diverse contents: foods, beverages, oil, chemicals, etc. [8]. Food is made up to heterogeneous chemical content like significant amount of water, organic acid, salt, nitrate etc. First of all tin and iron, which might affect the organoleptic quality of food as well as lead to accelerate the corrosion. If lacquer coated cans get scratched somewhere, it change the organoleptic attributes and self-life of food. Consequently, that bare inside the container lead to oxidation reactions of tin with food substances [9]. The pH value played a major effect. At pH above two the tin found in form tin hydroxide [Sn (OH)2] with low solubility. In another side, there are complex forms of tin appears with some chemicals like alcohols, esters, fatty acids and constant with some organic acids [10]
During the preservation of canned citrus juices, an interaction occurs between the components of the canned food and the material of the can. Corrosion in canned acid products is frequently influenced by the chemical composition of the products, character of the tin plate and the presence of the corrosion accelerators such as sulfites, sulfur dioxide and oxygen [11]. Studies of corrosion mechanism have shown that the process involves dissolution of the tin coating and steel base with evolution of hydrogen [12].
Scully (2000) recorded that a variety of methods such as electrical resistance, gravimetric-based mass loss, quartz crystal microbalance- based mass loss, electrochemical, and solution analysis methods enable the determination of corrosion rates of metals [13].

Food is canned in either plain tinplate or lacquered tin free steel. Although some food processing industries prefer to use lacquered tin plate which is very expensive but offers a longer shelf life. The quality of can is a major factor in the operations of food processing industries. Tin is considered to be quite resistant to attack by organic acids but undergoes corrosion if oxygen is present in the solution. In such cases, the food product may pick up corroded tin, which will affect the color and taste. With prolonged exposure, it may also have toxic effect on the quality of the food [14].

Tinplate container for liquid food packaging is extensively use due to most of its advantages:

- These containers are made with plain carbon steel plates with thin tin-coating.
- Ease of packing and sterilizing.
- Anaerobic environment of the sealed can.
- Transporting and minimizing the loss of vitamin potency in food stuff.

It is essential that the use of additives in canned food has to be carefully considered with respect to their performance and the reactions will take place between metal surface and present chemicals. These additive in electrolyte solution are detrimental to the anti-corrosion performance of tinplate because a reversal of cathodic/anodic behavior was noticed in galvanic couple between low carbon steel and tin and as a result the tin coating is no longer acts as a sacrificial anode and rapid corrosion of tinplate occurs.

Dissolution of tin into food:
Tin can enter foods either from natural sources, environmental pollution, packaging material or pesticides. Higher concentrations are found in processed food and canned foods. Dissolution of tin depends on the following factor [15]

- the food matrix,
- acidity,
- presence of oxidizing reagents (anthocyanin, nitrate, iron and copper)
- presence of air (oxygen) in the headspace ,
- time and
- temperature

FSA (2002) studied by testing of 1200 fully lacquered cans and found that decreased of 99.5% below the United Kingdom limit of 200 mg/kg of tin [16].
Corrosion occurs in different metals after a certain amount of time. Corrosion usually is caused by air particles getting into a small pore of the metal. It is a typical process that is not always preventable [17].
Tinplate corrosion depends on many factors:

1. Can material (tin coated steel, tin free steel)
2. Nature of the organic coating (epoxy, polyester, acrylic resins)
3. Enamel properties (adhesion, porosity and corrosion resistance)
4. Nature of contacting media (aqueous, fatty foodstuffs)
5. Composition of the contained product (acid foods, sulfur and salt etc.(Barilli et al.,2003; Montanari et al., 1996) [18,19]

Recently SEM in combination with EDS have proven to be a powerful and effective tool to investigate the many causes of corrosion in metal food containers [20, 21, and 22]. By using scanning Kelvin probe and scanning acoustic microscopy the lacquer failure has also been studied [23]

2. Material and Sample collection

Tinplate is a low carbon steel product with thicknesses between 0.17 and 0.53 mm [24] tinplated in electrolytic bath; thus combines the strength and malleability of steel with the corrosion resistance and weldability of tin [25,26]. Tinplate is mainly used in food packaging; ideal for this purpose because it is not toxic, light, strong and chemically stable [27]. The resulting coating has the following layered structure from bottom to top begins with an alloyed FeSn2 layer that protects the steel against galvanic corrosion by oxidizing species; and a tin layer which provides durability when acts as a sacrificial anode [28].
Tinplate is a thin low-carbon mild steel sheet, cold reduced, coated on both sides with pure electrolytic tin, used for food packaging. Study was conducted in two parts (A & B). Study of part A was conducted at our lab by preparing the corrosive media in the laboratory. Study of part B was conducted at Tata Steel R&D lab with SEM equipped with an energy dispersive X-ray (EDX) technique.

2.1. Sample Collection

Two type of sample collected for the experiment. Collected sample were made of lacquered and non-lacquered using food grade lacquer.

1. a) One sample of 50x50 mm size of equally tin- coated with 5.6/5.6 gm /m^2 of pure tin and Non-lacquered.
   b) One another sample of 50x50 mm size, equally tin-coated with 5.6/5.6 gm/m^2 of pure tin and Lacquered with food grad lacquer (epoxy phenolic).
2. a) Sample of 50x50 mm size of equally tin-coated with 2.8/2.8 gm /m^2 of pure tin and non-lacquered.
   b) Another sample of 50x50 mm size of equally tin- coated with 2.8/2.8 gm/m^2 and lacquered with food grade lacquer (epoxy phenolic).

Table 1: Chemical composition

Elements	Sample 1	Sample 2
C	0.079	0.070
Mn	0.41	0.66
Si	0.014	0.016
P	0.020	0.007
S	0.019	0.012
Cu	0.010	0.009
Ni	0.009	0.007
Cr	0.009	0.017
Mo	0.017	0.008
Al	0.059	0.038
Fe	Rest	Rest

2.2 Corrosive Media

Tomato juice solution prepared with citric acid (10 gm/lit) and sodium chloride solution (13 gm/lit). pH of the solution was regulated 3-4 by addition of Sodium hydroxide.

2.3 Experimental Method for Part "A" study

This study was made at our laboratory by total immersion of test specimen in prepared corrosive media (tomato juice solution) and kept for two weeks at room temperature.
After two weeks the sample was removed from the solution, cleaned, dried and check the weight. The corrosion rate was calculated by weight loss method as per formula given by Terence Bell (precedent & founder of strategic metal investment Ltd., editor of Strategic metal report).

Corrosion rate (mm/y) = 87.6 x (W/DAT)
Where:
W = weight loss in milligrams.
D = metal density in g /cm^3
A = area of sample in cm^2.
T = time of exposure of the metal sample in hours
87.6 = constant factor

2.3.1 Test Method

Tinplate sample coated with 2.8/2.8 gm/m^2 and with 5.6/5.6 gm/m^2 weighed prior to experiment and hanged in two individual glass container with the help of nylon thread. Heat up the tomato juice solution up to 70 deg. C and poured in both the glass container so that the sample completely deep into the solution. Closed the container with air tight cap and kept at room temperature (25±2°C) for two weeks.

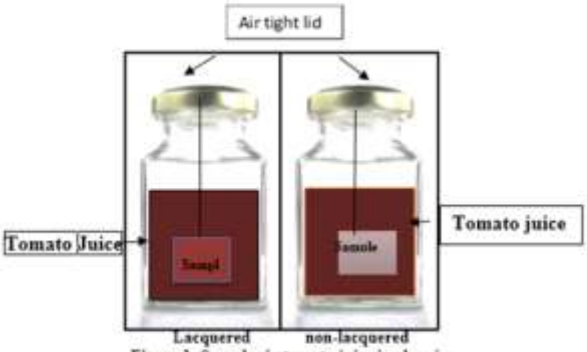


Figure 1: Samples in tomato juice in glass jar

After two week (336 hours), open the glass container, removed the sample from the tomato juice, cleaned with water, dried and weighed on electronic balance. Corrosion rate was calculated from the weight difference obtained from the experiment.
It was noticed that weight difference was obtained in non-lacquered sample. No weight difference was observed in the lacquered sample after completion of lab experiment.

Corrosion rate (mm/y) = 87.6 x (W/DAT)
Where:
W = weight loss in milligrams = 5.2718 – 5.2642
= 0.0076 gm = 7.6 mg
D = metal density in g /cm^3 = 7.23
A = area of sample in cm^2 = 50x50 mm2 = 25 cm^2
T = time of exposure of the metal sample in hours
= 336 hours (2 weeks)
87.6 = constant factor
Corrosion rate for non-lacquered sample calculated as
Corrosion rate (mm/y) = 87.6 (7.6/25x7.23x336)
= 0.011 mm/y

Non-lacquered tinplate (coating wt. 5.6 gm/m²)		**Lacquered tinplate (coating wt. 5.6 gm/m²)**	
Before Expt.	**After Expt.**	**Before Expt.**	**After Expt.**
			
Non-lacquered tinplate (coating wt. 2.8 gm/m2)		**Lacquered tinplate (coating wt. 2.8 gm/m2)**	
Before Expt.	**After Expt.**	**Before Expt.**	**After Expt.**
			

Figure 2: Photograph of sample before and after experiment

Photograph shows that 5.6 gm/m2 non-lacquered tinplate sample not withstand in the tomato juice, coated tin about 3/4th coated tin have been dissolved in the juice. Lacquered tinplate sample have no any corrosion effect and successful withstand with the juice.
Photograph of Tinplate of 2.8 gm/m2 coating sample non-lacquered also not withstand and almost all coated chromium dissolved in the solution. Lacquered Tinplate sample should not be failed in tomato juice but during experiment it was observed that lacquered sample failed due to corrosion.
Visual observation of 2.8 gm/m2 tin-coated sample surface indicated the pitting like corrosion effect on the lacquered surface. To study the failure of lacquering, the sample is being examine by Scanning Electron Microscope (SEM) coupled with Energy Dispersive X-ray spectroscopy (EDS).

2.3.2 Experimental Method for Part "B" study

Study was made by Scanning Electron Microscope coupled with Energy Dispersive X-ray and Auger Electron Spectroscopy analysis at Tata Steel R&D laboratory.
The morphology of non-lacquered tinplate shown in the following photograph.

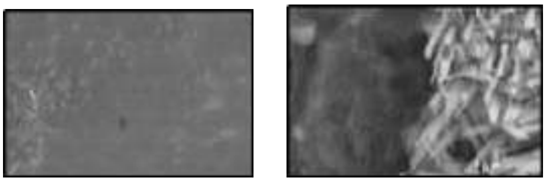

(a) **(b)**
Figure 3: Non-lacquered tinplate morphology

Figure (a) represent the surface before the experiment and figure (b) represent the surface after completion of two week experimental period. It shows the stick-shaped corrosion product on the surface.

The failed lacquered tinplate sample in corrosion test was examined by SEM along with EDS and Auger Electron Spectroscopy (AES) technique to know the reason of failure in corrosion test.
For this a fresh sample of 50x50 mm was collected from the remaining part of the lacquered tinplated sheet used for corrosion test. The sample surface was examined and found that tinny eye holes were present on the lacquered surface. Sample was treated with boiling mixture of Aniline and Ammonium hydroxide to remove the lacquer layer. Sample cleaned, dried and examine under microscope which revealed tinny eye holes were associated with small white spots. These eyeholes in the lacquer were also detected in the non-lacquered tinplate.
Examination of lacquered sample was studied in following sequence.

I. SEM examination
II. SEM examination of de-coated tin from the sample surface.
III. Examination of rougher portion as observed in SEM examination with Auger Electron Spectrometry (AES).

Observations during above examinations are as follows:

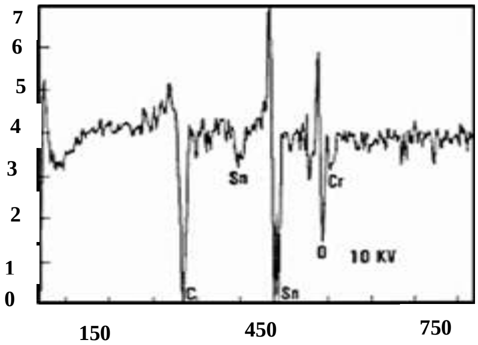
- The first step of SEM examination revealed that the white spots were the area of cleaved (split) tin shown in figure 4.
- SEM examination in second step was done on the same sample after de-coating of tin from the surface with the help of Clark's solution (2 gm antimony oxide in 100 ml HCL). It was observed that the surface beneath the cleaved tin found rougher than the surroundings.
- To examine this rougher portion, we use the Auger Electron Spectrometer (AES). The obtained spectrum from AES revealed an abnormal amount of carbon in the pores of the cleaved area as shown in figure 5. Whereas no carbon was detected in the adjacent to un-cleaved area as shown in figure 6.

Secondary electron micrograph of lacquered tinplate sample shown below:

Figure 4: Cleaved tin (pitted area)

Auger Spectrum (No. of Electrons vs Electron Kinetic Energy)

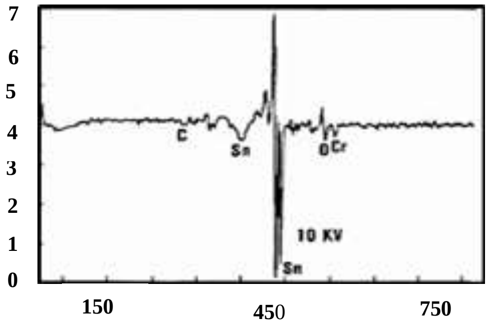
Electron no.



Kinetic Energy eV

Figure 5: Presence of carbon in cleaved area

Electron no.

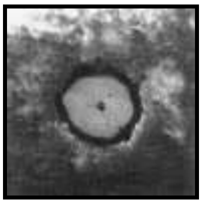


Kinetic Energy eV

Figure 6: Normal tinplate surface

SEM examination also revealed the eye holing problem (Fig.7) of lacquer, occurred due to ruptured micro blisters in the lacquer and appeared to be a case of solvent popping (Fig.8). The flow lines in the lacquer film and incipient blisters have observed at high voltage SEM examination as shown in fig.9

Secondary Electron Micrograph

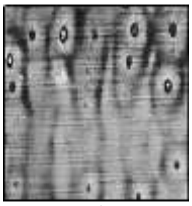


Eye hole in Lacquer Acceleration Voltage 20 kV

Figure:7

Blister in Lacquer Acceleration Voltage 12 kV

Figure:8



Lacquer flow lines with incipient blister Acceleration Voltage 32 kV

Figure: 9

It was also noticed during SEM examination that there were many small particles around the perimeter of the eyehole. To know the particle nature, it was anlaysed through EDS, which gave indication that the particle contained sodium and chromium which was most probably originated from sodium dichromate treatment solution in tinning line. X-ray spectrum generated by EDS analysis from the entire scan area of SEM shown in figure 10.

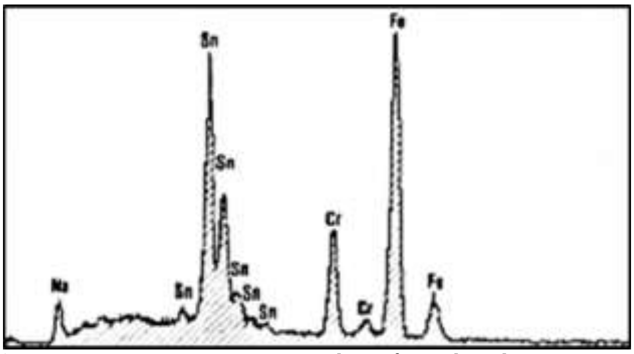
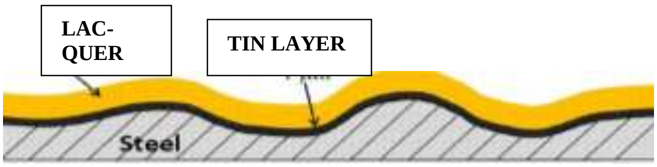


Figure 10: Energy Dispersive X-ray analysis of Particle, indicating Presence of Sodium di-chromate

2.3.3 Corrosion Mechanism

Figure 11 shows the corrosion mechanism for tinplate coated with food grade lacquer film in the corrosive solution tomato jounce contains NaCl electrolyte solution.

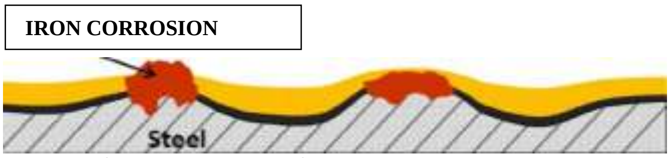


Defect free coated Lacquer with constant thickness & perfect adhesion

Figure 11(a)

Lacquer coating discontinuity & less thickness in rougher areas

Figure 11(b)



Corrosion first occurs beneath of lacquer coating then corrosion product, Break the film and visible directly.

Figure 11(c)

Figure 11

2.4 Discussion

- Figure 2 shows the tinplate after 336 hours of immersion in tomato juice solution shows preferential points which created corrosion and corrosion products. However, the tin coating was 2.8/2.8 gm/m2 and if it was defects free then it protect iron longer but observed red corrosion of the iron in the experiment, most probably Tin layer has discontinuities and defects.

- Figure 3 shows the non-lacquered tinplate morphology. It shows the stick-shaped corrosion product on the surface after the experiment over.

- Figure 4 shows the cleaved tin (pitted area) on the surface. This is the area where molten tin was not able to wet the steel sheet surface as a result no tin coating was available on that area.

It is only possible when a foreign material was present on the steel surface. To detect these foreign material sample was examined with Auger electron spectrometer (AES)

- Figure 5 & 6 shown the Auger spectrum, no. of electrons vs Electron Kinetic energy which was obtained by the above sample examination with AES. The obtained spectrum indicated the presence of an abnormal amount of carbon in the pores of cleaved areas of tin-coating. Whereas no carbon was detected in the adjacent to the cleaved area of tin.

- Figure 7 shows the lacquer eye holing problem. It was a lacquering process problem and obtained due to rupture of micro blister in the lacquer and looks like a case of solvent popping shown in figure 8.

- Figure 9 showed the lacquer flow line with incipient blister when examined the sample under higher voltage with SEM. At higher voltage, flow lines in the lacquer film and incipient blisters could be observed over a good portion of the plate. These conditions indicated that the problem was related to the surface tension of the lacquer, its solvents, and temperature gradients in the curing oven.

- Figure 10 shows the X-ray spectrum generated by EDS analysis from the entire scan area of SEM. During observation at higher magnification many small particles around the perimeter of the eyehole were observed. To know the particle nature, it was anlaysed through EDS, which gave indication that the particle contained sodium and chromium which was most probably originated from sodium dichromate treatment solution in tinning line.

- Figure 11 shows the corrosion mechanism. Figure 11(a) shows the Defect free coated Lacquer with constant thickness & perfect adhesion, figure 11(b) for Lacquer coating discontinuity & less thickness in rougher areas and figure 11(c) shows the Corrosion first occurs beneath of lacquer coating then corrosion product break the film and visible directly.

2.5 Conclusion

1. The results showed that the ferrous base exposed at the defects (anode). The food grade lacquer was able to retard the corrosion of the tinplate but in experiment it was failed and corrosion occurred at lacquered surface of tinplate coated with 2.8/2.8 gm/m2 tin.

2. Furthermore, the corrosion products showed that the corrosion occurs at the rougher area (irregularities) arising from defects in the steel base. By increasing the exposure time, the electrolyte (juice) permeated the tinplate substrate through lacquer cracks and defects in the steel base. First, corrosion occurs in the areas of Lacquer coating discontinuity & less thickness in rougher area, then corrosion products break the film and are visible directly.

3. The results obtained by the experiment showed that the lacquered sample had a good performance when immersed in the juice containing sodium chloride. The corrosion was detected few days of immersion with a small pit. After two week (336 hours) the experimented sample displayed preferential points which created corrosion and corrosion products around them.

4. Based on the results, we found that the lacquering on tinplate is effective as a protective coating in corrosive media when the steel base is free from defect as shown in the experiment. Any pores and thickness variation in lacquer coat is detrimental to the base metal. Lacquer retards the action of corrosion and ensures the integrity of the tinplate for longer when exposed to corrosive media.

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(Sourajyoti Dey)

Date:

Place: Jamshedpur, India

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