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Effects of Corona Treatment on Surface of Aluminum Foil for Retort Pouch

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Abstract: Retort pouch is one kind of flexible packaging which is made from plastic film or aluminum foil laminating together. Generally, retort pouches will use adhesive to combine each layer of film or foil together. If the lamination between each layer is not good enough, the delamination of each layer might occur. Therefore, the effects of corona treatment on surface of aluminum foil for retort pouch (PET/Al/CPP) were studied. This research was aimed to compare bond strength ability of Al/CPP layer, seal strength, and tensile strength of laminated film (PET/Al/CPP), both before and after sterilization. Moreover, it was aimed to compare the shelf life of laminated film (PET/Al/CPP) that held ketchup and tuna for a period of 7, 14, 21, and 30 days. There were five samples used in this experiment, the laminated film without corona treatment on Al surface and the laminated film with corona treatment on Al surface at 2, 2.5, 3, and 4 kW. The results showed that the laminated film with corona treatment (in all levels) on aluminum surface had higher bond strength, seal strength, and tensile strength than the laminated film without corona treatment on aluminum surface. The results of the shelf life of laminated film (PET/Al/CPP) that held ketchup and tuna paste for a period of 7, 14, 21, and 30 days showed that the bond strength ability of the film without corona treatment on aluminum decreased and the laminated film was delaminated on Al/CPP layer. In a way, the bond strength ability of laminated film with corona treatment (in all levels) on aluminum slightly decreased. It was also indicated that the level of voltage on corona treatment did not statistically affect bond strength ability of Al/CPP layer, and seal strength and tensile strength of laminated film (PET/Al/CPP).

Keywords: *Corona treatment, retort pouch, sterilization, Aluminum foil*

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I. INTRODUCTION

In the past, humans used natural materials, such as leaves, to use as package in order to protect and maintain the quality of products. Later, the packaging was developed not only to protect and maintain the quality of products but also to gather the products into the units for ease of movement or transportation. Recently, packaging has been evolved into many aspects including; glass, wood, paper, metal, and plastic. Thus, the role and function of packaging have also increased [1, 2]. Nowadays, packaging has a higher growth potential and retort pouch is a kind of packaging which is classified in the category of flexible packaging. Retort pouch is a flexible packaging which is formed into a pouch and sealed after filling products inside. Retort pouches are strong and resistant to heat and pressure during commercial thermal sterilization [3]. Retort pouches are made from various plastic films or aluminum foil to be combined together to provide the desired properties, for example; low gas (oxygen) and moisture permeability, low hydrophilic properties, heat sealable and sterilisable, resistant to penetration by fats, oil and other food components, etc., [4]. Generally, retort pouches are used for high sterilized products, such as ready-to-eat food products and the products that required extended shelf life. Normally, the products inside retort pouches can be stored at room temperature.

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Moreover, retort pouches are light weight. Therefore, they can reduce transportation and storage cost. There are various styles of retort pouches, and the pouches also can be printed graphics directly. Since retort pouches are easy to form and easy to open. Therefore, both packaging industries and consumer sectors are increasingly focusing on retort pouches [5].

Apparently, mechanical properties and barrier permeability properties of plastic are lower than metal and glass. Then, it is necessary to use various types of plastic films to make retort pouches. Each type of plastic film has its unique properties. Therefore, retort pouch manufacturers have to combine it together in order to have optimal oxygen and water vapor transmission permeability for maintaining quality of the products inside. In a way, the mechanical properties in terms of strength and heat resistance of retort pouches need to be able to resist temperatures up to 135 degrees Celsius during sterilization process.

Mostly, retort pouches are made from Polyethylene Terephthalate (PET) film, aluminum foil, and Polypropylene (PP) film [6]. All these 3 materials are combined together into laminated film by an adhesive in the lamination process. Each layer of the retort pouch has its own function as follows:

1. Main structure layer is directly in contact with food. It has the thickest structure with high heat resistance and high strength to withstand the pressure in retort process. Thus, it must be able to seal with heat but not react with food inside packages.

2. Barrier material layer is a medium layer that protects moisture, light, and gases. The most popular use is aluminum foil and sometime, nylon may be used to increase the strength of the pouches. However, the use of aluminum foil will make the retort pouches not be able to use in microwave ovens. Then, SiOx or AlxOy are used to coat retort pouches for protecting the permeability and changing the pouches to be microwavable pouches.

3. The outer layer must have ability to print graphics and data easily and well, but it also needs to be durable and not easy to tear [2].

Since retort pouches are made of various types of plastic film and aluminum foil attached to each other by using adhesive as a binder. If the films or aluminum foils are poorly bonded, the pouches might eventually peel off and then the protective properties of the pouches will definitely reduce or not function at full capacity. Each of the above-mentioned problems is a real problem in the production process of some manufacturing companies in Thailand. The frequency occurred problem is the delamination of film after the pouches have passed the sterilization process. The delaminated pouches could not maintain the quality of the products from manufacturers to consumers and they would cause faster deterioration or rapidly spoil products inside the pouches. In addition, not only the fact that the delamination pouches could not store and extend the shelf life of the products properly, but the delamination pouches also could exacerbate the appearance of the packaging and decrease the value of goods.

As problems mentioned above, the researchers recognized the importance of the impact of problems of film delamination of retort pouches. Then, they were interested to increase the adhesion ability of each film or aluminum layer. Corona discharge treatment is one of the methods that can enhance adhesion ability between film and foil. The corona treatment increases surface energy of film or aluminum foil layer by introducing polar group on the surface of film or foil. Therefore, the treated substrate surface will have better wetting and adhesion abilities [7, 8, 9, 10]. This study was in collaboration with Hutamaki (Thailand) Co., Ltd., by studying the effects of corona treatment on the surface of aluminum foil for retort pouch (PET/Al/CPP). The research was aimed to compare bond strength ability of Al/CPP layer, and seal strength and tensile strength of laminated film (PET/Al/CPP), both before and after sterilization. Moreover, it was aimed to compare the shelf life of laminated film (PET/Al/CPP) that held ketchup and tuna for a certain period.

II. EXPERIMENTAL

A. *Materials*

1. PET (Polyethylene terephthalate) 12 micron/Al (Aluminum foil) 9micron/CPP (Cast Polypropylene) 70 micron without corona treatment on Al surface.

2. PET 12 micron/Al 9 micron/CPP 70 micron with 2 kW. corona treatment on Al surface.

3. PET 12 micron/Al 9 micron/CPP 70 micron with 2.5 kW. corona treatment on Al surface.

4. PET 12 micron/Al 9 micron/CPP 70 micron with 3 kW. corona treatment on Al surface.

5. PET 12 micron/Al 9 micron/CPP 70 micron with 4 kW. corona treatment on Al surface.

B. *Instruments*

1. Thickness Gauge

2. Laminating Machine (Sung An Machinery Co., LTD.)

3. Sealing Machine (Tester Sangyo Co., LTD)

4. Tensile Tester (Instron Co., LTD)

5. Autoclave (Sanoclav Co., LTD)

C. Procedure

1) Bond strength testing: The specimens from 5 laminated film samples were prepared by cutting them along their machine direction to 15 mm. x 150 mm. for 10 pieces. Delaminated Al. layer from CPP layer more than 5 cm long and clamped it on tensile tester. The test was tested according to ASTM F904-98. All samples were tested for both before and after sterilization.

Fig. 1. Bond strength testing on tensile tester

2) Seal strength testing: Prepared specimens from 5 samples of laminated film and cut them along machine direction to 15 mm. x 150 mm. for 10 pieces. Then sealed them with heat sealer at 200°C, 3 Pascal, for 3 seconds and then tested by Tensile tester. The test was tested according to ASTM F88 (both before and after sterilization).

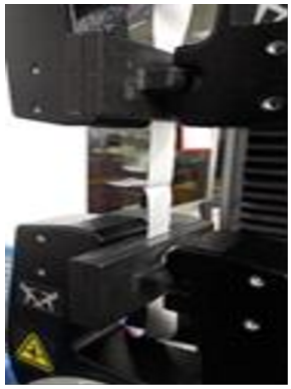

Fig. 2. Seal strength testing on tensile tester

3) Tensile strength testing: Prepared specimens from 5 samples of laminated film and cut them along machine direction to 15 mm. x 150 mm. for 10 pieces. All specimens were tested by tensile tester according to ASTM D882 (both before and after sterilization).

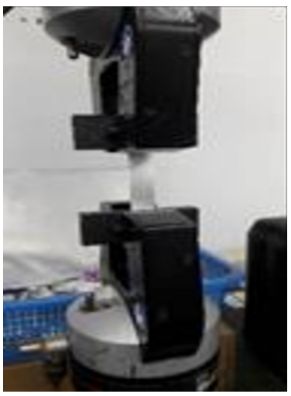

Fig. 3. Tensile strength on tensile tester

4) Evaluating shelf life of packages: Constructed retort pouches from 5 samples of laminated film. The dimensions of the pouch were 95 mm. Œ 125 mm. Œ 25 mm, and then 70 gram of ketchup and wet cat food (Tuna paste) were separately packed and sealed into pouches. Then, they were sterilized at 125 degrees Celsius for 45 minutes. Later, all ketchup and wet cat food (Tuna paste) were kept in a control room at 45-50 degrees Celsius. All the pouches were placed horizontally with the front of pouch faced up in order to let the products inside equally contact with the packages. After that, the samples were taken out every 7, 14, 21, and 30 days to wash, clean, and then observe. Then, the pouches were cut along their machine direction to 15 mm. x 150 mm. and tested for their bond strength according to ASTM F904-98. All the data were statistically analyzed by SPSS program (Statistical Package for the Social Science for Windows).

III. RESULTS AND DISCUSSION

A. Bond Strength Test

From Table 1, the data showed that bond strength of Al/CPP laminated film for both before and after sterilization was significantly different at 0.05 confident level. The data indicated that bonding strength of the laminated film without corona treatment before and after sterilization were 6.21 and 5.55 kg/15 mm, respectively.

TABLE 1
BOND STRENGTH OF FIVE TYPES OF AL/CPP LAMINATED FILM, BOTH BEFORE AND AFTER STERILIZATION

Sample	w/o corona	Corona 2.0 kW.	Corona 2.5 kW.	Corona 3.0 kW.	Corona 4.0 kW.
Bond strength (kg/15mm)	Max load ± SD	Max load ± SD	Max load ± SD	Max load ± SD	Max load ± SD
Before sterilization	6.21 ± 0.36	6.61 ± 0.12	6.71 ± 0.28	6.5 ± 0.10	6.53 ± 0.10
After sterilization	5.55 ± 0.60	6.51 ± 0.11	6.66 ± 0.21	6.56 ± 0.10	6.46 ± 0.12

Meanwhile, the results of bonding strength of Al/CPP laminated film with corona treatment on Al surface at 2, 2.5, 3, and 4 kW for both before and after sterilization were not significantly different at 0.05 confidence level. In a way, bond strength of laminated film without corona treatment on Al surface was significantly lower than bond strength of laminated film with corona treatment on Al surface at all energy levels.

According to the data of bond strength of Al/CPP laminated film in machine direction, it indicated that non-corona treated film on aluminum surface before and after sterilization had less bond strength than corona film on aluminum surface.

In addition, the adhesion of non-corona treated film on the aluminum surface was decreased after sterilization. While the corona treated film on the aluminum surface had higher bond strength, and adhesion value did not decrease after sterilization. Generally, corona treatment has charged high energy to the material's surface.

Then, its surface is filled with electron or charged atoms. Those electrons or charged atoms will oxidize and give more polar surface to the material. Then, the adhesion on the surface is increased which made the adhesion between each layer stronger and thicker [11]. Thus, the corona energy level did not affect the adhesion of the film. The results of this study were consistent with the research on surface energy retention on plastic films with corona treatment to improve film adhesion for gravure printing [12].

B. *Seal Strength Test*

From Table 2, the data showed that seal strength of the sealant of all laminated film was significantly decreased after sterilization. The laminated films with the highest to the lowest sealing strength before sterilization were corona treated film at 4 kW, corona treated film at 3 kW, corona treated film at 2.5 kW, corona treated film at 2 kW, and non-corona treated film on aluminum surface, respectively. While the laminated films with the highest to the lowest seal strength after sterilization were corona treated film at 2.5 kW, corona treated film at 3 kW, corona treated film at 2 kW, corona treated film at 4 kW, and non-corona treated film on aluminum surface, respectively. Thus, it was also implied that the increasing of corona treatment energy did not affect the increasing of seal strength of all Al/CPP laminated films.

TABLE 2
SEAL STRENGTH OF FIVE TYPES OF AL/CPP LAMINATED FILM, BOTH BEFORE AND AFTER STERILIZATION

Sample	w/o Corona	Corona 2.0 kW.	Corona 2.5 kW.	Corona 3.0 kW.	Corona 4.0 kW.
Seal strength (kg/15mm)	Max load $\pm$ SD	Max load $\pm$ SD	Max load $\pm$ SD	Max load $\pm$ SD	Max load $\pm$ SD
Before sterilization	47.5 $\pm$ 4.38	52.6 $\pm$ 2.19	54.9 $\pm$ 1.46	55.5 $\pm$ 2.10	56.2 $\pm$ 2.35
After sterilization	35.8 $\pm$ 2.85	45.4 $\pm$ 1.44	45.9 $\pm$ 0.98	45.5 $\pm$ 1.97	44.9 $\pm$ 1.92

According to the results from the heat sealing properties (at 200 ^{0}C, 3 Pascal for 3 seconds) test in accordance with ASTM F88, it showed that seal strength of all 5 laminated films had decreased after sterilization. Thus, the corona treated on aluminum surface laminated film still exhibited stronger sealing strength both before and after sterilization than non-corona treated film. Since during the sterilization process, some heat vapor could pass through the film layer and reduce the seal strength. These results were relatively consistent with the research on the effect of heat during sterilization process on strength of flexible packaging (pouch) which indicated that the heat from the process statically and significantly decreased the seal strength of pouches [13].

C. *Tensile Strength Test*

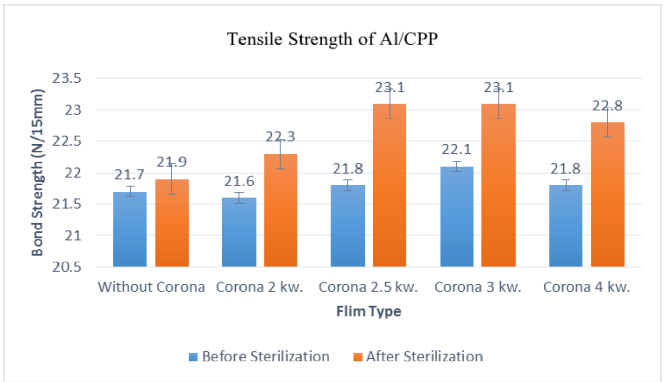

Fig. 4. Tensile strength of five types of Al/CPP laminated film, both before and after sterilization

From the tensile strength test of all Al/CPP laminated films in machine direction according to ASTM D882, the results showed that the non-corona treated film and the corona treated at 2 kW on aluminum surface laminated film had no difference in tensile strength both before and after sterilization. While the corona treated on aluminum surface laminated films at 2.5, 3, and 4 kW had higher tensile strengths than those without corona treated and corona treated at 2 kW laminated film. Moreover, it was also indicated that tensile strength of all types of Al/CPP laminated film was increased after sterilization since the sterilization process @ 125 ^{0}C for 45 minutes had affected polypropylene film, a semi-crystalline polymer, by inducing stress and relocating of polymer molecules. Thus, in the slow cooling-down stage during sterilization, polymer molecules of PP have more time to orientate or relocate, then the chances of crystallization are greater. Therefore, the tensile strength of all types of laminated film was increased after sterilization. In addition, the corona treatment on aluminum surface of Al/CPP laminated film also provided better adhesion; as a result, tensile strength of the corona treated on aluminum surface film has increased [14]. Shelf life of laminated film (PET/Al/CPP) that held ketchup and tuna paste for a period of 7, 14, 21, and 30 days.

TABLE 3
BOND STRENGTH OF FIVE TYPES OF AL/CPP LAMINATED FILM THAT HELD KETCHUP FOR 30 DAYS

Sample	w/o Corona	Corona 2.0 kW.	Corona 2.5 kW.	Corona 3.0 kW.	Corona 4.0 kW.
Bond Strength (kg/15mm)	Max load $\pm$ SD	Max load $\pm$ SD	Max load $\pm$ SD	Max load $\pm$ SD	Max load $\pm$ SD
Day 7	7.01 $\pm$ 0.21	6.83 $\pm$ 0.16	6.93 $\pm$ 0.18	6.97 $\pm$ 0.22	7.12 $\pm$ 0.35
Day 14	6.98 $\pm$ 0.17	7.00 $\pm$ 0.16	7.11 $\pm$ 0.23	7.21 $\pm$ 0.22	7.35 $\pm$ 0.29
Day 21	3.61 $\pm$ 0.96	6.67 $\pm$ 0.33	6.47 $\pm$ 0.11	6.69 $\pm$ 0.54	6.76 $\pm$ 0.16
Day 30	1.30 $\pm$ 1.30	6.83 $\pm$ 0.16	6.93 $\pm$ 0.13	6.0 $\pm$ 0.70	6.17 $\pm$ 0.62

From Table 3, the data showed that the bond strength values of all five laminated films on day 7 were not significantly different. On day 14, bond strength values of non-corona treated film and corona treated film at 2.0 kW. were significantly different from the corona treated film at 3.0 kW., and 4.0 kW. Moreover, the corona treated film at 2.5 kW. was significantly different from the corona treated at 4 kW. at 0.05 confidence level.

According to the result, it was also found that on days 21 and 30, the bond strength value of the non-corona treated film on the aluminum surface was the lowest decreased when compared to all laminated films. Though, the appearance of the laminated film was only slightly delaminated since polypropylene has an acid-insoluble property [2], but it could be seen that stains of ketchup still permeated into the film layer.

This was possibly due to the adhesives used in laminating process that could not resist acid from ketchup. In addition, the pouches were kept in a conditioned room at 45-50 ^{0}C, at this quite high temperature; it could result in an increase in acidity in ketchup and eventually ketchup could penetrate through the film layer to the adhesive layer [15].

While corona treated on the aluminum surface films exhibited better adhesion between film layers. Therefore, it was more difficult for acid to permeate into film layers.

From Figure 5, it was found that laminated film without corona treatment on aluminum surface had delamination of film layer. There were delaminated lines and stains of ketchup that penetrated into film layer.

It is indicated in the “Flexible retort pouch defects identification and classification Manual” that delamination outside the sealed area of the pouch could affect the seal strength of the pouch [4]. Therefore, the bond strength of the non-corona treated film was decreased.

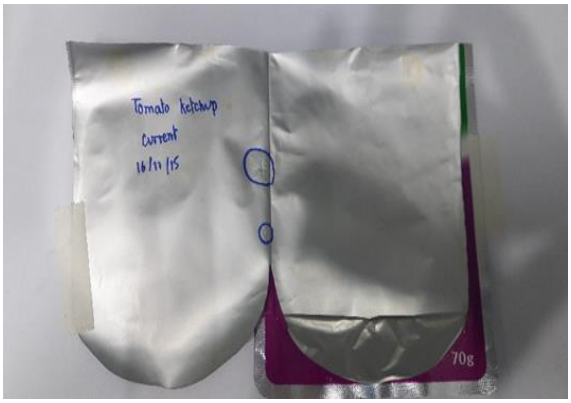

Fig. 5. Laminated film without corona treated on aluminum surface that held ketchup for 30 days

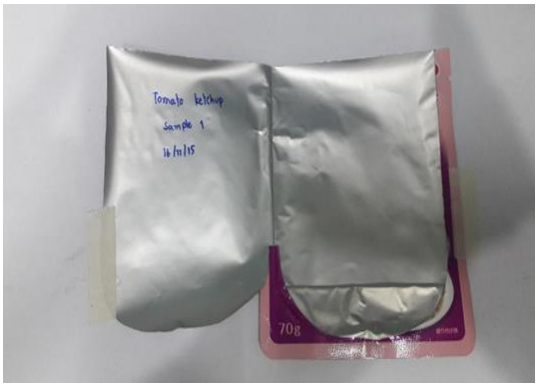

Fig. 6. Laminated film with 2.0 kW. corona treated on aluminum surface that held ketchup for 30 days

From Figure 6, it showed that the laminated film with 2.0 kW. corona treated on aluminum surface had no delamination of film layer and no stain of ketchup between film layers.

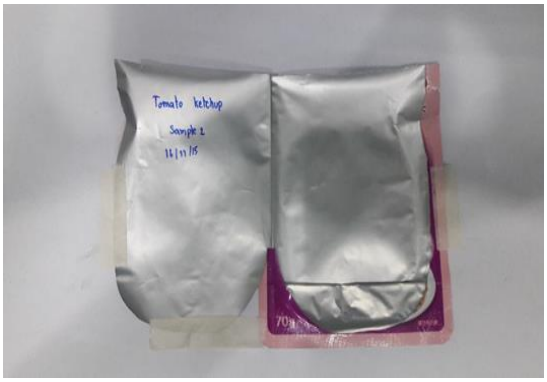

Fig. 7. Laminated film with 2.5 kW. corona treated on aluminum surface that held ketchup for 30 days

From Figure 7, it showed that the laminated film with 2.5 kW. corona treated on aluminum surface had no delamination of film layer and no stain of ketchup between film layers.

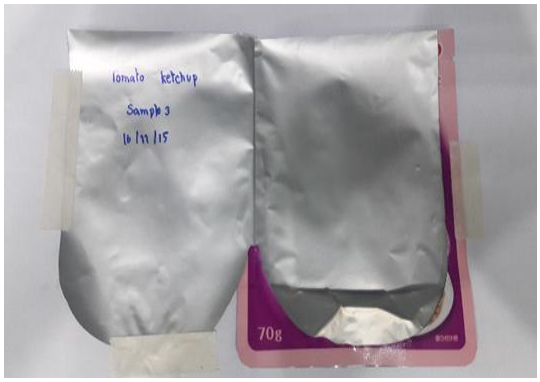

Fig. 8. Laminated film with 3.0 kW. corona treated on aluminum surface that held ketchup for 30 days

From Figure 8, it also showed that the laminated film with 3.0 kW. corona treated on aluminum surface had no delamination of film layer and no stain of ketchup between film layers.

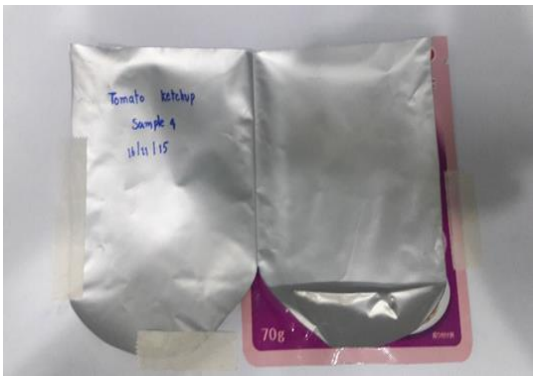

Fig. 9. Laminated film with 4.0 kW. corona treated on aluminum surface that held ketchup for 30 days

From Figure 9, it showed that the laminated film with 4.0 kW. corona treated on aluminum surface had no delamination of film layer and no stain of ketchup between film layers.

TABLE 4
BOND STRENGTH OF FIVE TYPES OF AL/CPP LAMINATED FILM THAT HELD KETCHUP FOR 30 DAYS

Sample	w/o Corona	Corona 2.0 kW.	Corona 2.5 kW.	Corona 3.0 kW.	Corona 4.0 kW.
Bond Strength (kg/15mm)	Max load ± SD	Max load ± SD	Max load ± SD	Max load ± SD	Max load ± SD
Day 7	6.04 ± 0.54	6.15 ± 0.34	6.01 ± 0.38	6.20 ± 0.16	6.07 ± 0.16
Day 14	5.84 ± 0.52	6.08 ± 0.32	6.07 ± 0.18	6.10 ± 0.29	6.04 ± 0.13
Day 21	5.67 ± 1.73	6.06 ± 0.27	6.04 ± 0.16	6.06± 0.24	6.01 ± 0.15
Day 30	2.33 ± 0.28	5.16 ± 0.87	5.80 ± 0.89	5.45 ± 1.04	5.72 ± 0.69

From Table 4, the data showed that the bond strength values of all five laminated films on day 7 were not significantly different. On day 14, the bond strength of laminated film without corona treatment was significantly decreased and it also had the lowest bond strength on day 21 as well as on day 30. Thus, it also indicated

that on day 30, the bond strength values of all types of laminated films were significantly decreased if compared with the bond strength values on day 7. It was also indicated that laminated film with corona treated at 2.5 kW. on day 30 had the highest bond strength. It might be implied that the higher level of corona treatment energy on aluminum surface did not provide the better adhesion or higher bond strength to laminated film.

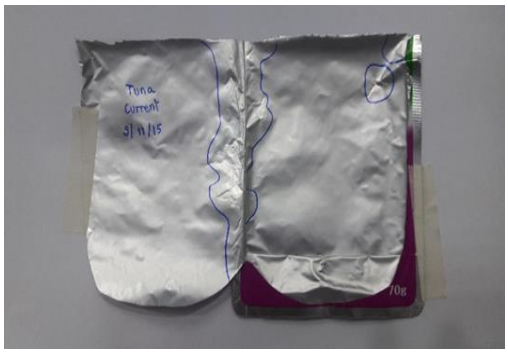

Fig. 10. Laminated film without corona treated on aluminum surface that held tuna paste for 30 days

From Figure 10, it showed that the laminated film was delaminated until aluminum layer could not attach to CPP layer.

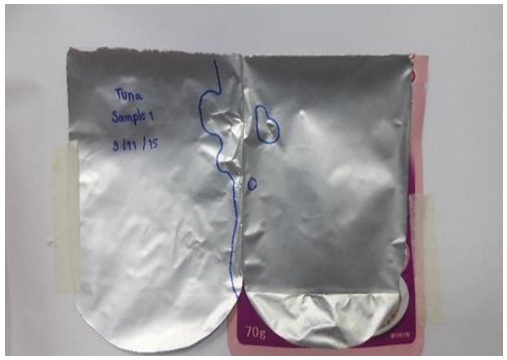

Fig. 11. Laminated film with 2.0 kW. corona treated on aluminum surface that held tuna paste for 30 days

From Figure 11, it showed that the laminated film was slightly delaminated but overall area of laminated film still had proper adhesion.

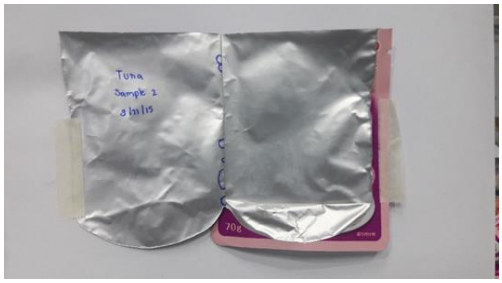

Fig. 12. Laminated film with 2.5 kW. corona treated on aluminum surface that held tuna paste for 30 days

From Figure 12, it was found that laminated film with 2.5 kW. corona treated on aluminum surface was delaminated with fine line marking on the rim of the pouch.

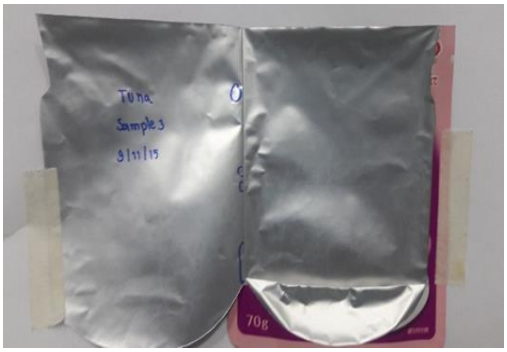

Fig. 13. Laminated film with 3.0 kW. corona treated on aluminum surface that held tuna paste for 30 days

From Figure 13, it was also found that laminated with 3.0 kW. corona treated on aluminum surface was delaminated with fine line marking on the rim of the pouch.

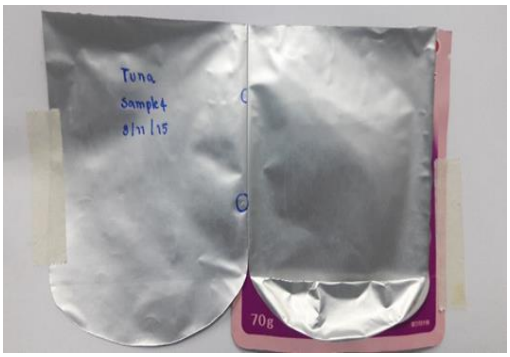

Fig. 14. Laminated film with 4.0 kW. corona treated on aluminum surface that held tuna paste for 30 days

From Figure 14, it was found that the laminated film with 4.0 kW. corona treated on aluminum surface was very slightly delaminated.

According to the results of bond strength test of all types of laminated film that held tuna paste for 30 days, the data showed that non-corona film on aluminum surface and laminated film with 2 kW. corona treated on aluminum surface had the highest decrease in bond strength values, respectively. There was delamination between aluminum and CPP layer on those types of laminated film. While the bond strength values of laminated film with 2.5, 3, and 4 kW. corona treated on aluminum surface were slightly decreased. It was found that all the laminated films were slightly delaminated with fine lines on film's surface. Since polypropylene film cannot resist to fatty acid absorption well from tuna paste, therefore, when CPP film was not literally attached to aluminum,

the fat inside tuna paste would permeate through the film layer and swell the aluminum layer [2]. In addition, in the "Adhesion of Aluminum Foil to Coatings-Stick with it", it was indicated that the fat could cause the delamination, leading fat-filled void, flaws, and cavities [16]. However, the laminated films with 2.5 - 4 kW. corona treated on aluminum surface exhibited better bonding behavior. Therefore, it was more difficult for the fat in tuna paste to penetrate through the film layer.

IV. CONCLUSION

According to the results, it could be concluded that the laminated film without corona treated on aluminum surface, both before and after sterilization, had less bond strength than the laminated film with corona treated on aluminum surface at all energy levels. In addition, the bond strength of non-corona treated on the aluminum surface film was reduced after sterilization. While the corona treated on aluminum surface laminated film had higher bond strength, and its bond strength value did not decrease after sterilization.

It also could be concluded that seal strength of laminated films was decreased after sterilization. Thus, the corona treated on aluminum surface laminated film still exhibited stronger sealing strength, both before and after sterilization, than non-corona treated on aluminum surface laminated film. Moreover, non-corona treated on aluminum surface laminated film and corona treated laminated film at 2 kW. had no difference in tensile strength, both before and after sterilization. While the corona-treated laminated films at all energy levels had higher tensile strength than those without corona treatment and corona treated at 2 kW. laminated film. It was also found that tensile strength value of all corona treated on aluminum surface laminated films was increased after sterilization.

It was also indicated that corona treatment on aluminum foil surface for retort packaging could provide more bond strength between aluminum and CPP layer and longer extent shelf life of the products in retort packaging. If the manufacturers want to produce retort pouches with other types of film or want to use the pouches to fill other types of products, more testing should be carried out before use. Thus, the energy level of corona treatment should be properly selected to suit the selected film.

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