

Ozone, generated at the corona treater, as an adhesion promoter in extrusion coating

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ABSTRACT: *The trials documented in this paper were run on a pilot coextrusion coating line at the Tampere University of Technology's Institute of Paper Converting in Finland. The study was conducted to test the ozonization system that was installed in our pilot line to treat the polymer melt in extrusion coating. The effect of the ozone, generated at the corona treater, on adhesion was studied. Ozone was first captured and transported to the nip with separate pipes. It was then led to an air knife near the air gap and blown against the polymer melt. The measured adhesion showed the usefulness of this technique. The following parameters were varied to determine the effectiveness of the ozone: substrate, line speed, coating weight, and melt temperature. Results indicated that thicker coating weights and higher melt temperatures improved adhesion values. The corona-generated ozone clearly improved adhesion compared to corona-treated or untreated samples.*

KEYWORDS: *Adhesion, corona discharge, extrusion coating, kraft papers, low density polyethylene, ozone, supercalendered papers, surface treatment.*

Several mechanisms can affect extrusion coating adhesion: wetting, mechanical interlocking, diffusion, and primary and secondary bonding. Adhesion is primarily promoted by the secondary bonding,

when the oxidized surfaces, containing polar groups, are pressed together at the laminator nip (1). The methods used to oxidize the web (or to increase its polar groups) include applying primers and flame and co-

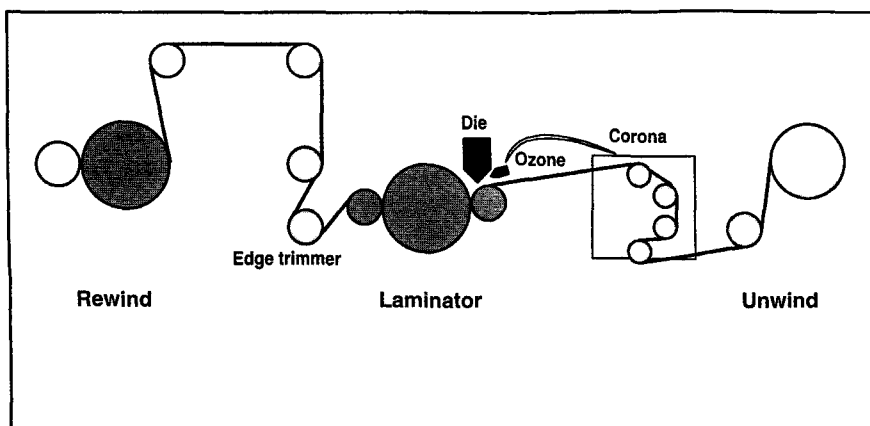
rona treatments. On the other hand, the polymer can be oxidized by additives, melt temperature, and ozone treatment.

Typical corona-treating equipment consists of a high-frequency generator, a high-voltage transformer, and a treater station assembly that includes a dielectric-covered, grounded roll and an electrode. Higher frequencies are most suitable for corona treatment, because they are quiet and give uniform treatment to the rapidly moving web. High voltages are used to ionize the air. The buffer or dielectric roll coating prevents discharge sparks which cause pinholes and adds resistance to the circuit to assure a uniform corona along the length of the electrode. The treater station is usually enclosed to restrain the ozone generated in the corona bursts (2).

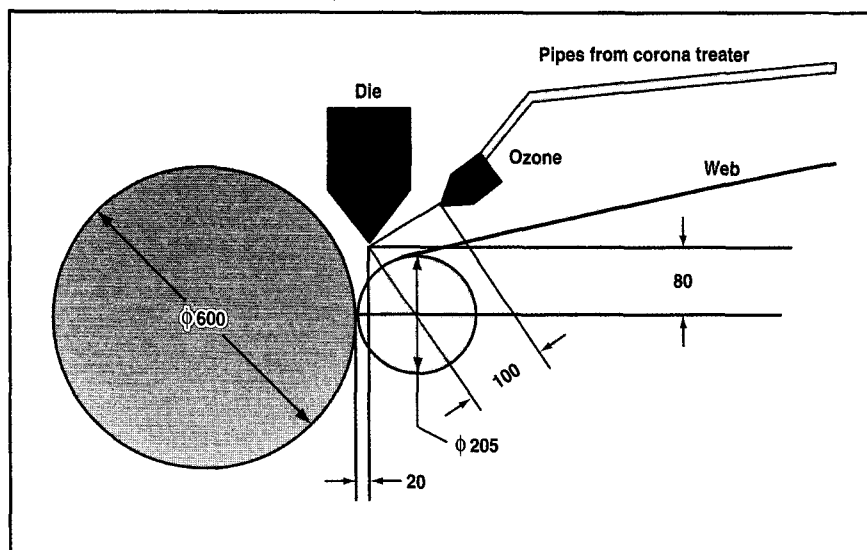
In corona operation, the air gap between the electrode and the moving web surface has a lower dielectric breakdown voltage than does the web. Because of the applied high-frequency voltage, the air in the gap becomes ionized and forms a gaseous conductor that is a bluish corona. The corona burst generates the following species of oxidants: ozone, atomic oxygen, oxygen-free radicals, and high-speed electrons. The high-speed electrons accelerated during the corona burst reach an energy level of about 10 eV, which is sufficient to break the bonds present on

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1. Line operation.



2. Die lip position vs. laminator nip and position of ozone treater. Dimensions are in mm



most substrate surfaces. The broken bonds result in highly reactive free radicals on the surface. Therefore, the surface is oxidized, leading to the formation of polar groups (primarily hydroxyl, carbonyl, and amide) (2).

Low-density polyethylene (LDPE) is a nonpolar polymer, and therefore it must be oxidized, normally by high melt temperatures or ozone treatment, to improve adhesion in extrusion coating. The melt oxidizes in the air gap to form polar groups on the melt surface. Oxidation is a time- and temperature-dependent chemical reaction. The time is determined by the air gap and line

speed, and the maximum temperature is limited by the product quality requirements such as odor, taste, and heat sealing and blocking (low-molecular-weight products on the surface). Ozone preferentially oxidizes the melt on the substrate side, which allows adequate adhesion and minimizes the undesirable oxidation effects on the chill-roll side. A further benefit is that the air gap can be reduced, leading to improved melt stability and lower neck-in (3).

In this study, we first captured the ozone (generated at the corona treater), transported it to the nip, and then blew it against the polymer melt, using the normally wasted oxidizer in the system.

Procedure

Extrusion coating

In our trials, we used a pilot coextrusion coating line consisting of three extruders and a 350-mm-wide, five-layer vane die. Only one extruder with a polyethylene-type screw was used. The line layout is shown in Fig. 1, and Fig. 2 presents the configuration of the pretreatment units (also note the pipes used for ozone transport).

Constants during the experiments were as follows: chill roll diameter = 600 mm, kraft paper chill roll temperature = 20°C, SC paper chill roll temperature = 25°C, die lip opening = 0.5 mm, air gap = 8 cm, nip durometer = 90 Shore A, and nip pressure = 30 kPa. Three line speeds (30, 60, and 90 m/min), two screw speeds (20 and 40 rpm), and three melt temperatures (280°C, 300°C, and 315°C) were used in the trials.

Two webs were used in the trials: kraft paper (80 g/m² bleached; Wisakraft/Kymmene, Finland) and one-side-coated supercalendered wood-free special paper (70 g/m² Simcastor/United Paper Mills, Finland). The back (noncoated) side of the wood-free paper, referred to in this study as SC paper, was extrusion coated. The coating polymer was a 0.918-g/cm³, 7.5-MFI LDPE (NCPE 7518/Borealis, Finland).

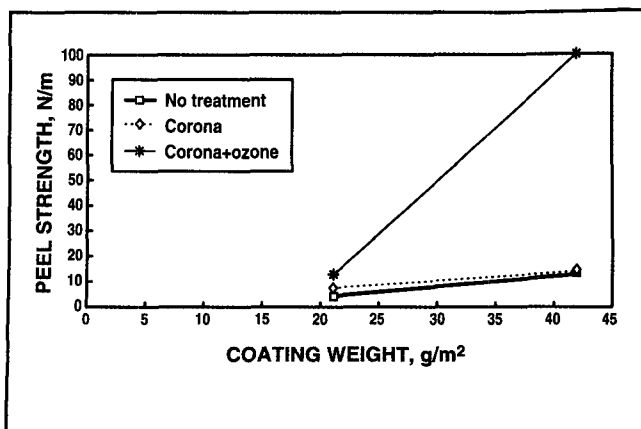
Pretreatments

The treater station in our corona unit (Vetaphone ET 3-3.5 MX) consists of four rubber-covered rolls and knife electrodes, and the generated ozone was transported through pipes to oxidize the polymer melt. A corona power of 1.5 kW was kept constant during the trials. Three variables were used: no pretreatment, corona on, and both corona and ozone on.

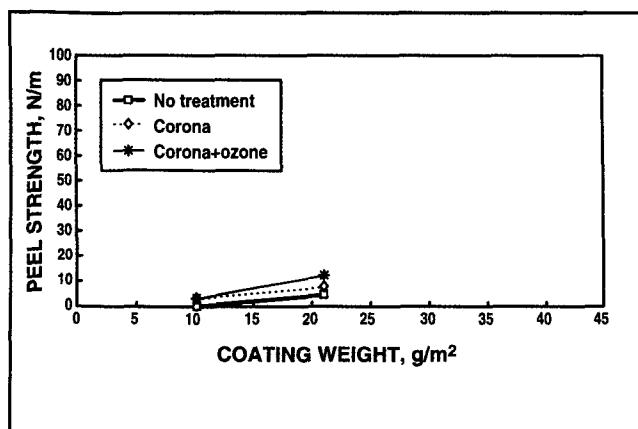
Analytics

The adhesion was first evaluated by hand. If peeling caused fiber tearing

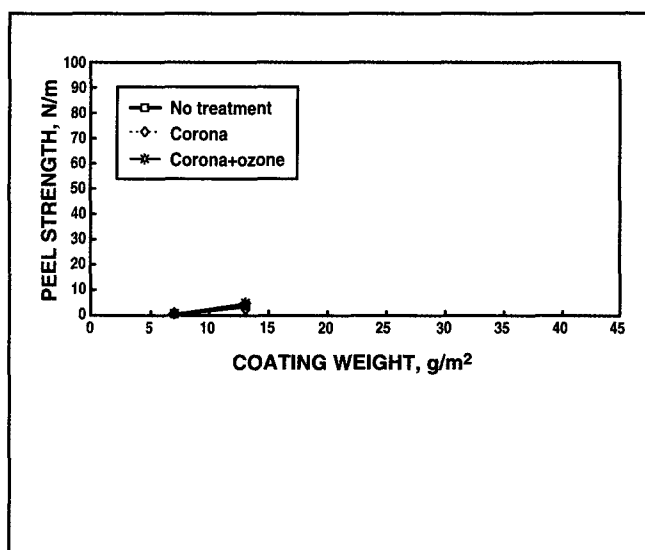
3. Kraft paper/LDPE at 280°C and 30 m/min



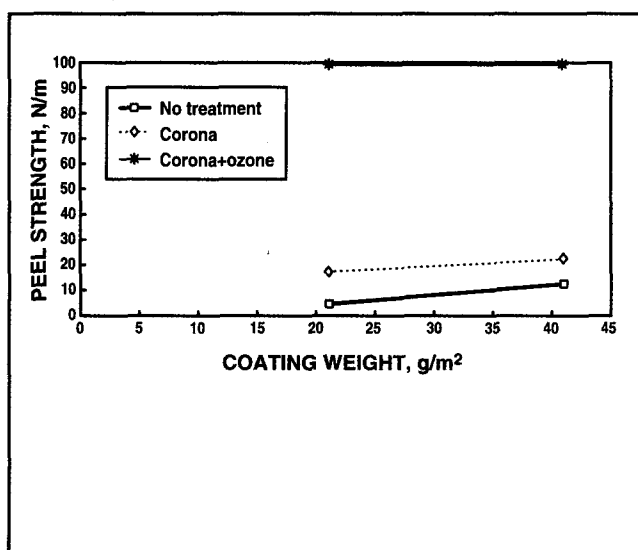
4. Kraft paper/LDPE at 280°C and 60 m/min



5. Kraft paper/LDPE at 280°C and 90 m/min



6. Kraft paper/LDPE at 300°C and 30 m/min



in the substrate, the sample was given a value of 100 N/m (see Figs. 3–14). Otherwise, the peelable samples were measured according to ASTM Standard D1876 (T-peel test).

Results

Kraft paper

Test results for the kraft paper samples are shown in Figs. 3–8. At 280°C, the melt temperature adhesion was poor, regardless of the pretreatment, below 20 g/m² coating weight. Untreated and corona-treated webs showed poor adhesion at each weight. The only good (fiber

tear) result was achieved with a corona/ozone-treated 42-g/m² sample.

At 300°C, the melt temperature adhesion level was generally better than at 280°C. However, the untreated and corona-treated webs again showed poor adhesion at each coating weight. At 30 m/min line speed, the 21-g/m² and 42-g/m² samples pretreated with corona/ozone showed good adhesion. On the other hand, the 21-g/m² sample, run at 60 m/min, showed much poorer adhesion (31 N/m).

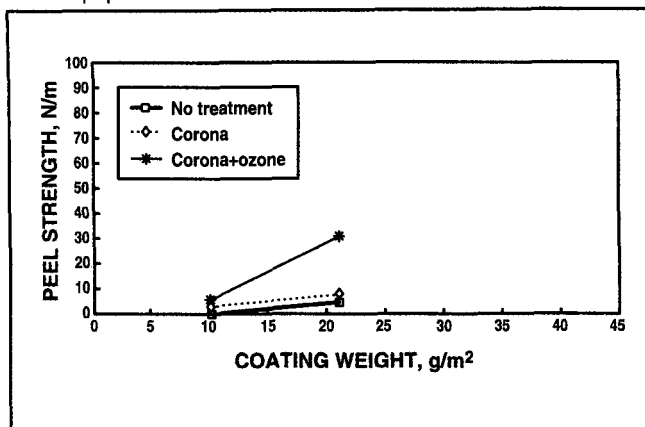
SC paper

Results for the SC paper samples are shown in Figs. 9–14. At 280°C

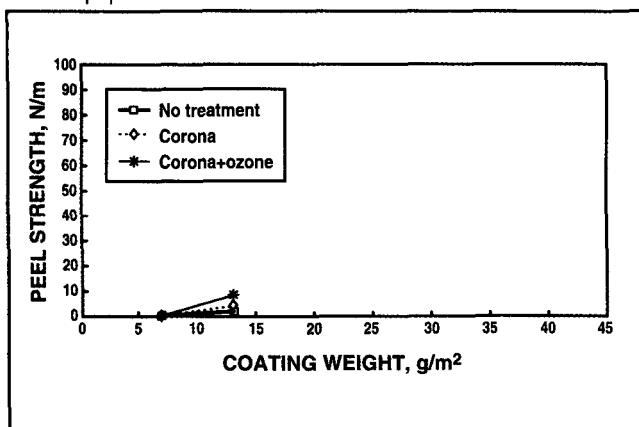
melt temperature, all of the corona/ozone treated samples showed good adhesion. The untreated webs showed poor adhesion at each weight. Corona-treated, 15- and 27-g/m² samples showed good adhesion at 30 m/min line speed. However, at 60 m/min, the adhesion was poor at the 7 and 12 g/m² weights.

At 300°C melt temperature, all of the corona/ozone-treated samples again showed good adhesion. The untreated webs showed poor adhesion at each coating weight. Corona-treated, 15- and 29-g/m² samples exhibited good adhesion at 30 m/min line speed. However, at 60 m/min, the adhesion was poor at the 8 and 15 g/m² weights.

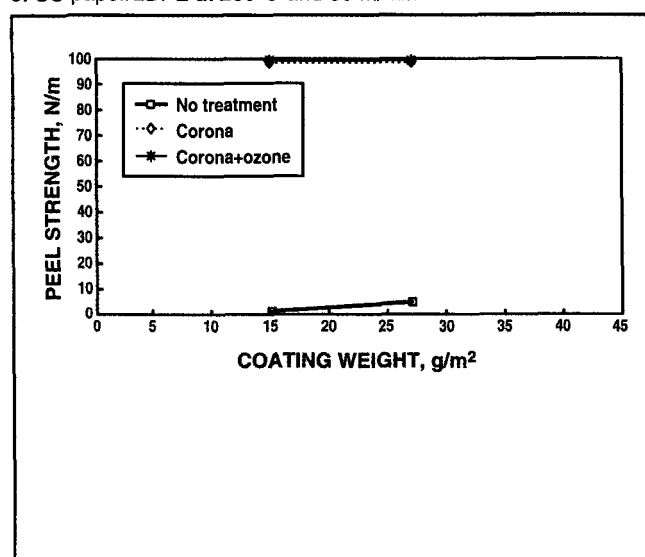
7. Kraft paper/LDPE at 300°C and 60 m/min



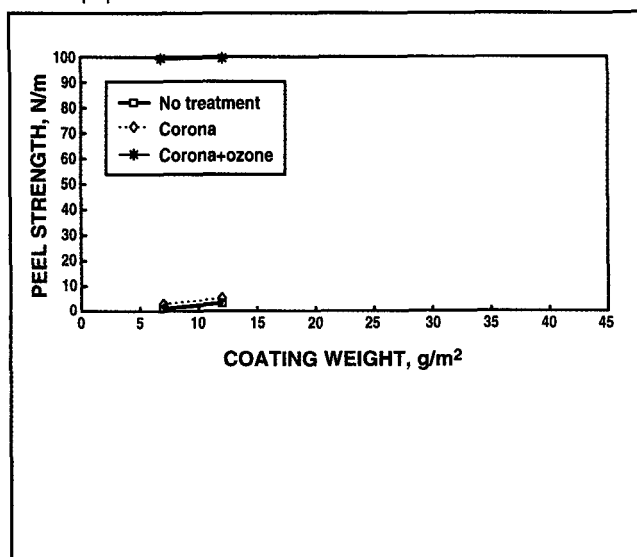
8. Kraft paper/LDPE at 300°C and 90 m/min



9. SC paper/LDPE at 280°C and 30 m/min



10. SC paper/LDPE at 280°C and 60 m/min



At 315°C melt temperature, the corona/ozone-treated samples showed good adhesion. Untreated webs showed poor adhesion at each coating weight. However, in contrast to the results run at lower melt temperatures, the adhesion of each corona-treated sample was good.

Analysis

Pretreatments

The untreated samples showed poor adhesion in every case. Even though, at the 315°C melt temperature, the polymer is capable of strong bonding (oxidation), the lack of similar

oxidation characteristics on the web surface prevents adequate adhesion.

On the other hand, corona treatment oxidizes the web surface; this results in better adhesion values in most cases compared to the untreated samples. At 315°C, both the melt and corona-treated web surfaces promote bonding, and therefore the adhesion in these samples was good. With SC paper at 280°C and 300°C melt temperatures, line speed was the major factor in achieving good adhesion; at 30 m/min the adhesion was good, but at 60 m/min it was poor. This is probably due to the longer oxidation time of the melt at lower line speeds. The longer pen-

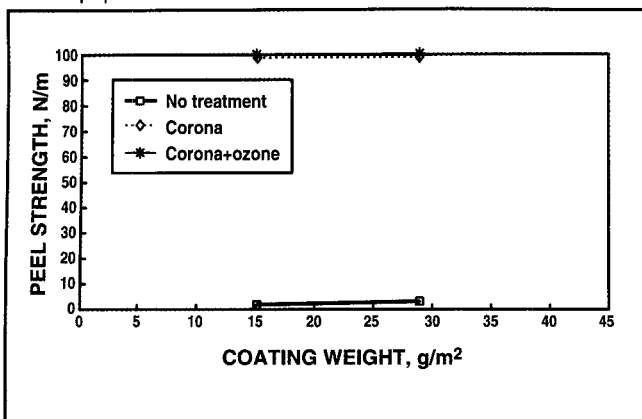
etration/bonding time at the nip contact point also promotes adhesion.

Corona oxidizes the web surface and ozone the melt surface resulting in a perfect combination (in theory) of pretreatments, because both surfaces are therefore attractive (polar) to form bonds. This is readily apparent in the results of these trials; the corona/ozone-treated samples clearly had the best adhesion values, especially with the SC paper.

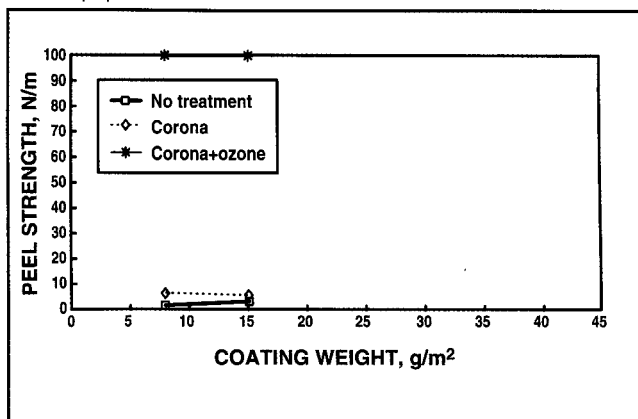
Substrate

It is difficult to compare the adhesion results of these two substrates because of the different chill roll temperatures, different contact areas

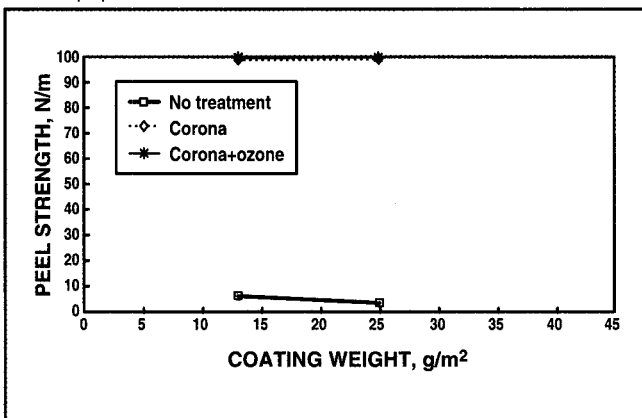
11. SC paper/LDPE at 300°C and 30 m/min



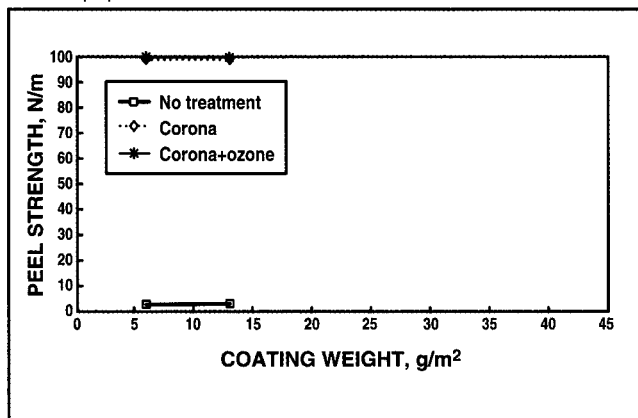
12. SC paper/LDPE at 300°C and 60 m/min



13. SC paper/LDPE at 315°C and 30 m/min



14. SC paper/LDPE at 315°C and 60 m/min



(surface roughness) between the web and the polymer, and other physical and chemical differences on the substrate surfaces. However, the results clearly show that the SC paper displayed better adhesion at similar conditions.

Summary

This study was conducted to test a system in which ozone, generated at the corona treater, was captured and transported to the nip and was blown against the polymer melt to oxidize the film. The effect of the oxidation was evaluated by measuring the adhesion of extrusion-coated samples produced at different conditions.

The results clearly show that it is advantageous to oxidize both the web (e.g., corona) and melt (ozone or melt temperature) surfaces to achieve good adhesion in extrusion coatings. If only one of the surfaces is oxidized, the adhesion is usually inadequate. If neither surface is oxidized, poor adhesion will probably result.

We have shown that it is possible to use the corona-generated ozone to oxidize the melt and thus improve adhesion. At this point, the effectiveness of this treatment at higher line speeds is not known. However, further studies at the Institute of Paper Converting will evaluate this variable. ■

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