

# Placement Report

Butlers Court Research & Development  
Arjo Wiggins

**Evaluation of surface treatment techniques for polypropylene and  
implementation of a method for testing ink adhesion**

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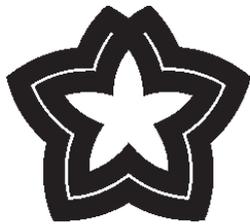
# EXAMENSARBETE, C-nivå

## Grafisk Teknik

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Titel Utveckling av ytbehandlingstekniker för polypropylen och implementation av en adhesion test metod		
Nyckelord Plast material, polypropylen, adhesion, korona, plasma, test metod.		

### Sammanfattning

När man använder plastmaterial som tryckbärare krävs en speciell ytbehandling för att man ska få ett bra och hållbart tryck. Vanligaste ytbehandlingstekniken för tillfället är koronabehandling. Denna ytbehandling har tyvärr visat sig att inte vara så hållbar i längden. Plasmabehandling som i detta fall använder sig av olika gaser vid behandlingen av polypropylen, visar sig i detta projekt vara mer effektivt i det långa loppet. När den plasmabehandlade ytan tryckts varar den goda kvaliteten på trycket under längre tid och adhesionen mellan tryckfärgen och ytan består. För att testa denna adhesion används för närvarande en standard (ASTM D3359). Denna standard har visat sig ostabil och beroende av många olika faktorer, som åstadkommer variationer i testresultaten. På grund av detta har nya testmetoder provats fram för att ett jämnare och mer tillförlitligt resultat vid test av adhesionen ska kunna ges.



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# DEGREE PROJECT

## Graphic Arts Technology

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Title Evaluation of surface treatment techniques for polypropylene and implementation of an adhesion test method		
Keywords Plastic material, polypropylene, adhesion, corona, plasma, test method		

### Summary

If a plastic material is used as a print bearer there are a need of a special surface treatment to get a good and durable printing. The most used surface treatment technique for the moment is corona treatment. This kind of treatment has unfortunately showed not to be so durable in the long term. Plasma treatment which in this case uses different kind of gases in the treatment of polypropylene is shown as a more effective treatment in this project. When the plasma treated surface has been printed is the good quality last much longer and the adhesion between the ink and the surface is remained. To test this adhesion is for the moment a standard used (ASTM D3359). This standard has appeared unstable and dependent at many different factors, which gives a big variation in the test results. Because of this has new test methods been carried out to give a more even and more reliable result in the test of the adhesion.

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# 1 Introduction

## 1.1 Structure of Arjo Wiggins

Arjo Wiggins is a company with assets mainly within two areas; in industry and in services. In the area of industry there is production of papers for creative applications, communication, images, security, packaging, medical and hospital use. Furthermore there are also production of carbonless paper and a wide range of industrial specialities. In the area of services the main application is the distribution of papers and supplies.

Arjo Wiggins is one of the world leading manufacturers of technical and creative papers present in Europe, America and Asia, with 7,500 employees worldwide. Some of their biggest customers are found within printing industries, design, publishing, advertising and paper industries.

One of the branches in the company is speciality products, which has 1,660 employees. In this area one of the brands is called Priplak. Priplak is a plastic product made of polypropylene which has gone through extrusion and calendering to become plastic sheets. This product will constitute the basis of this study.

## 1.2 Research and Development

There are three research- and development centres of Arjo Wiggins, two in France and one in Great Britain. These centres are involved in three areas of major importance to the group; breakaway innovation, development of new products and technical support.

Each site is equipped with experimental facilities, for example pilot plants, to be able to work with any necessary technical developments.

The R&D centre in Beaconsfield, England, is located just outside London in well-bred surroundings.

## 2 The project

### 2.1 Background

To use plastic material as a print bearer often causes a problem that has to be solved if a good print quality is to be received. The problem depends on the natural properties of the plastic material, such as low surface energy and polarity. These properties have to be increased so the ink sticks to the surface properly and remains there with a good appearance.

For example, a plastic material made of polypropylene has a surface energy of 32 dynes/cm (Dynes/cm = N/m). To be able to print with a lasting result on the surface, the surface energy has to be increased up to 45 dynes/cm.

### 2.2 Objectives

For ink to stick to plastic surfaces and remain there, the surface has to go through some kind of treatment. Through research and tests of plastic surfaces with different treatments the performance of the treatments will be evaluated.

The surface energy of the material can be increased by creating polar groups at the material surface. This will lead to better adhesion between the surface and the ink in as permanent condition as possible in matter of time.

The goal is to find the treatment that gives the best results of printing quality as well as long term print performance, without the deterioration of the bulk properties of the plastic.

The goal is also to find a treatment method that will provide the polypropylene with improved ink adhesion when compared to the currently corona treated product. The treatment must last over a period of three months without any loss of adhesion performance.

### 2.3 Method

There are several different methods to treat the unprinted, plastic surface in order to obtain better and longer lasting printability. Nowadays the most

usually used treatment is Corona treatment. Other treatments that are used are atmospheric plasma and low pressure plasma including chemical grafting. These treatments will be evaluated for their effectiveness on a polypropylene substrate.

The tests are accomplished using a DAT-equipment (Dynamic Absorption Tester) to find out if surface energy has been modified.

With the IGT-equipment, which is a method of laboratory printing, plastic samples are printed with UV-ink and afterwards they are tested with a specific adhesion test (ASTM D3359). This gives an indication on how well the ink sticks to the surface.

## 3 Plastic and printing

### 3.1 Plastic materials

Polymers are molecules that are very big. Naturally occurring polymers are for example polysaccharides and proteins. Synthetically produced polymers are the base of modern plastic materials. Examples of where polymers can be found in our everyday life are in paint, textiles, packaging, electronics, cars, house material and so on...

Furthermore, we as humans contain a lot of polymers and our hereditary factors are stored in a polymer. The daily food also contains a lot of polymers.

#### Thermosetting plastics

- Urea formaldehyde (UF)

One example of a thermosetting plastic is urea formaldehyde.

A thermoset plastic is a kind of plastic that can't be processed several times, i.e. it can not be melted and formed a second time. UF is a totally clear material that is favourable from a graphic point of view. It can be coloured without limits and is used for products that demand a resistance against for example fat.

Examples of products are containers and packaging for cosmetics, clean and hygienic. Another application is wet-strength paper. In this paper a UF-coating is formatted around the fibres which make the paper strong also in humid environments.

#### Thermoplastic materials

- Polyethylene (PE)

This is the most used and a versatile and proportionately cheap kind of commodity plastic. The material is resistant against most chemicals, does not absorb water and does not easily let through water steam which makes it a useful barrier material for example in milk containers. Polyethylene is easy to seal but difficult to glue. Other drawbacks of polyethylene are that it has low

tear resistance and lower exhaustion mechanics than for example polypropylene.

- Ethylene/vinyl acetate (EVA)

The main application for ethylene/vinyl acetate resins is hot melt adhesives and plastic film. The film has a good printability and is a very good barrier against gases and water steam; this makes it useful for provision-industry for food packaging.

- Polyvinyl chloride (PVC)

PVC in its pure form is a hard and fragile material that is not suitable for packaging. If a softening agent is added, a softer and more flexible material is produced.

The PVC-plastic is a very good barrier of gas which makes it a perfect material for packaging products with a high content of oil and fat. The material is also used within the pharmaceutical industry because of its good resistance against acids and alkalises.

The main application for PVC is bottles and containers and transparent film in cardboard boxes. The production of PVC is very cheap.

- Polystyrene (PS)

Polystyrene is a plastic with a relatively high resistance against gases, which makes the application of the material for cosmetics packaging to a good choice. PS is also used for different pots, for example yoghurt pots and disposable cups.

Within this application the properties of gas impenetrability and graphic appearance are very important. The film becomes almost shimmering and clear and is used for example in luxury window cardboard boxes. It is also possible to print the plastic with foil and other graphic effects.

Polystyrene is in the same price range as polyethylene, i.e. it is proportionately cheap.

- Cellulose acetate (CA) /crystalline cellulose

Cellulose acetate/crystalline cellulose is a hard, tough and fire safe plastic, which in the form of film is sparkling clear and transparent. It is often used as a plastic window in cardboard boxes. This is because of its brilliance and also of its good properties to be glued on cardboard. Furthermore the material can be found as a luxury wrapping around flowers.

- Regenerated cellulose

Regenerated cellulose is very clear and almost as transparent as glass. Uncoated, humidity sensitive film of cellulose is principally used for packaging that requires a high fat barrier and high permeability of water steam.

A good example of these products is pre-packed food to heat in the microwave oven. Coated, humidity insensitive cellulose film is used in packaging for cookies, crisps, sweets, nuts and so on.

- Polyethylene terephthalate (PET)

In the form of a film PET is very clear and tough. After the material has gone through a complicated treatment it gets even tougher and it also gets a good heat resistance. PET-film has a high tear-strength, bear repeated folding and has many good strength properties. The permeability of water steam is relatively low and the material is a good barrier of gas, odour and taste, and has a low absorption of water.

PET is for example used in packaging of vacuum for pre-packed food, coffee, and in provisions with a big content of fat. It is also a very common material in bottles for lemonade. If sheets of PET are used, the material is often first calendered to get a high glossiness. This is being done to get the sheets thermoformed and to be able to use it in transparent cardboard boxes. PET is also used in formed oven safe containers.

Unfortunately PET is approximately twice as expensive as polyethylene and polystyrene.

### 3.2 Printing techniques

The most common printing techniques used when printing on plastic are:

- Offset

Today the dominating printing method is offset printing. In this printing method almost every plane printed matter can be produced. When something is being printed with offset a plane printing plate is used, without difference in height. The original usually transfers via film to the plate, which then is developed.

Before the printing, the plate is moistened. The areas at the plate that would not emit ink during the printing attracts the water and leave the dry areas to attract the ink. The plate is pressed against a rubber covered cylinder which receives the ink and offset it on the paper. The plate is never in touch with the paper, thereby the name offset.

- Screen printing

In screen printing the printing form consists of a fine-meshed cloth through which the ink is pressed with the help of a rake. This makes the method fairly slow.

The advantage of screen printing is the possibility of printing on many different materials, such as on thick cardboard, plastic, metal, fabrics, wood and many other materials. The intensity and the covering of the pigments and the possibility to transfer thick layers of ink give good results on uneven and granular surfaces.

One drawback is that it is hard to control the ink quantity to a specific part of the picture. The registration between the colours is also less exact compared to the use of offset printing.

- Flexography

Flexography is actually a kind of letterpress printing technique which is being done in a web fed press of one or more colours. The printing is being done from blocks made of plastic or rubber.

Flexography is often used to print at plastic packaging, milk cartons, printing at wallpapers and even some newspapers are printed with this technique. One advantage of flexography is the high printing speed.

### 3.3 Ink adhesion

The ink adhesion depends on several different factors. The most important factors are:

- Wet-ability

Wet-ability is a concept that tells how well a liquid spreads (wets) over a solid surface. To measure this ability a so called DAT-equipment is used. The contact angle between the liquid and the substrate is measured and the wet-ability is in this way settled. If the material has a good wet-ability the contact angle is small. The contact angle is  $0^\circ$  if the wet-ability is perfect. If the wet-ability is poor the contact angle is big. A larger contact angle than  $90^\circ$  is possible. Water has a contact angle of about  $104^\circ$  on polyethylene. A low wet-ability occurs when a highly polar liquid, such as water, is contacted with a material of low or no polarity (e. g. polyethylene).

The DAT-test is measuring also the surface energy which is measured in dynes/cm. An untreated polypropylene sheet (substrate used for this study) has a contact angle greater than  $90^\circ$  and a surface energy of about 35 dynes/cm when tested with water. The ink does wet the substrate even at 33 dynes/cm, i.e. the print might appear good but the problem is that the ink will not adhere to the substrate.

- Stick-ability

The concept of stick-ability tells how well two materials stick to each other. For example, it can be a question of how well ink, glue or extrusion coated polymers stick to a given substrate.

### 3.4 Polypropylene (PP)

Polypropylene is a thermoplastic material which is chemically similar to polyethylene. The properties of the materials are also fairly comparable. Polypropylene is, together with polyethylene, the most used plastic in the packaging business.

The possibility to produce fibres is big, depending on that PP easily creates an orientated crystalline. Stretching the material under cold conditions gives a big increase of the crystallinity. The material gets unique properties of exhaustion and can resist a big number of folding, which is taken advantage of for products where folding is required after a line (hinge), such as for example, in plastic screens.

This plastic has a very good liquid- and gas barrier which also makes it a useful product for packaging of cookies, crisps, and other provisions that easily absorb and emit odour and are sensitive to the effect of oxygen. Another excellent property for this kind of plastic is a good sealing property.

Other applications for PP are for example households' articles, laboratory- and hospital articles for disposable use, containers, bottles, pipes, VVS-armature, fan-propellers, corrugated cardboard of plastic and plastic films.

The drawbacks of the material are that the UV-sensitivity is higher than for polyethylene. At lower temperatures, (20 °C) the material loses its impact strength. This can be avoided by copolymerisation with PE, which unfortunately lowers the stiffness of the material.

Polypropylene is at the same price level as polyethylene.

## 4 Surface treatments of plastic materials

### 4.1 Corona treatment

The process of corona implies that ionised air provoked by an electrical discharge of high voltage 10-50 kV treats the surface which causes a light oxidation of the material. The surface becomes more polar (with increased surface energy), which makes the ink adhere better to the surface. Surface activation is what is generally achieved with corona. The efficiency of the treatment is normally of a limited shelf-life.

However, recent developments have been using corona as a mean to activate the surface followed by the application of chemicals. This will lead to a reaction with the activated surface and further on modify its properties by chemical grafting or deposition. These processes show improvement over corona performance.

Advantages of corona treatment:

- Easily installed equipment
- Low expenses for the treatment

Drawbacks of corona treatment:

- Treatment of paper or fluorine polymers (polytetrafluoroethylene) is not possible
- Not a uniform, reproducible treatment: depends on temperature and humidity
- Contaminations can easily affect the quality of the treatment
- Corona treatment isn't so flexible because of the use of only one gas

### 4.2 Plasma treatment

When a plastic surface is treated with plasma the same principle is used as with corona treatment. The surface can be activated with an electrical ionisation of gas, but in this case a much lower voltage, and other gases than

air (e.g. He, Ar, O<sub>2</sub>, N<sub>2</sub>), are used. A soft indefinable cloud of ionised gas, without any visible electrical charge, is created during a plasma discharge.

Another difference between plasma- and corona treatment is that the bombardment of electrons is 100 times faster in plasma treatment, therefore allowing for a more efficient treatment. As with corona, surface activation is achieved, but chemicals can also be injected in the plasma gas to result in surface modification by chemical grafting/deposition.

There are two different treatment methods of plasma; Atmospheric plasma treatment (APT) and Low pressure plasma.

Advantages of Plasma treatment:

- Longer lasting treatment
- Possibility to treat thicker material
- Higher performance and durability for difficult materials, for example polytetrafluoroethylene.
- The treatment doesn't give any damage at the surface and the bulk properties are not altered.

## 5 Tests after surface treatment of plastic material

### 5.1 Surface characterisation

#### 5.1.1 Surface energy

The low surface energy of a plastic material surface depends on a lot of different factors. Hydrophilic (polar) properties are measured with water and the hydrophobic (dispersive) properties are measured with bromonaphthalene.

The Surface energy is the sum of the polar component energy and the dispersive component energy. In the case of polyolefins, such as PP, the polar component is very low. This does not promote adhesion as polar liquids will not wet properly the surface.

The surface energy has to be increased in order to get a plastic surface printable. To increase the surface energy and bring adhesion, the weak boundary layer (WBL) has to be removed.

WBL is a layer of water, oil or oxidation that contaminates the surface. When this weak boundary layer is removed a chemical reaction needs to be promoted, leading to an increased polarity (and thus to an increased surface energy). With a level of about 45 dynes/cm of the surface energy it is possible to print the plastic surface with a sufficient result.

To measure the surface energy a DAT-equipment (Dynamic Absorption Tester) is being used. Plastic samples of the substrates are prepared in stripes of 25 mm width. The computer software that is used is called DAT25Q.

These necessary settings are being done at the DAT-machine before starting measurement.

- Mode: 2
- Drop size: 4 µl
- Batch quality: 5
- Check time: 0.10 sec, 0.20 sec, 0.30 sec
- Timeout: 0.1 min.

When the measurement starts a high speed camera is filming the course of events during the test. The computer software calculates and gives the result in a table.

## 5.2 Evaluation of performance

### *5.2.1 Sample preparation*

#### *5.2.1.1 IGT-printing*

The first step in testing adhesion on a plastic material is to print samples at the IGT-printer, which is a laboratory printer. The samples are prepared and cut in 50 mm wide strips. They are printed at low speed and constant rolling.

An UV-ink is used, called Sicura Plast UV (black), which is dried at the lowest speed on a UV curing bench. The sample is printed with a pressure of 784 N and a print speed of 0.8 m/s.

#### *5.2.1.2 Optical density check*

After printing the optical density is checked. A densitometer is used to see if the density of the samples are within the limit of 1.6- 2.0. A density at 1.8 is the ideal value of the printed samples.

#### *5.2.1.3 Adhesion tests*

To test the adhesion between the substrate and the ink a standard test is being used (ASTM D3359). The samples have been split up in two parts. One part of the sample is left untouched 'Plain' and at the other part a specific grid pattern is being created 'Cross hatch'.

On both areas of the sample a special adhesive tape (Scotch Magic Tape 810) is carefully applied with a rubber. The tape is further ripped off at a constant speed and a specified angle.

Later on, an estimation of the level of the adhesion is being done. The level of adhesion on both areas (plain and cross hatch) is first estimated in a scale of 0-5, where 0 represents no adhesion and 5 represent full adhesion. The ink quantity is also specified in percentage of ink left at the surface, to give closer information of the result of the test.

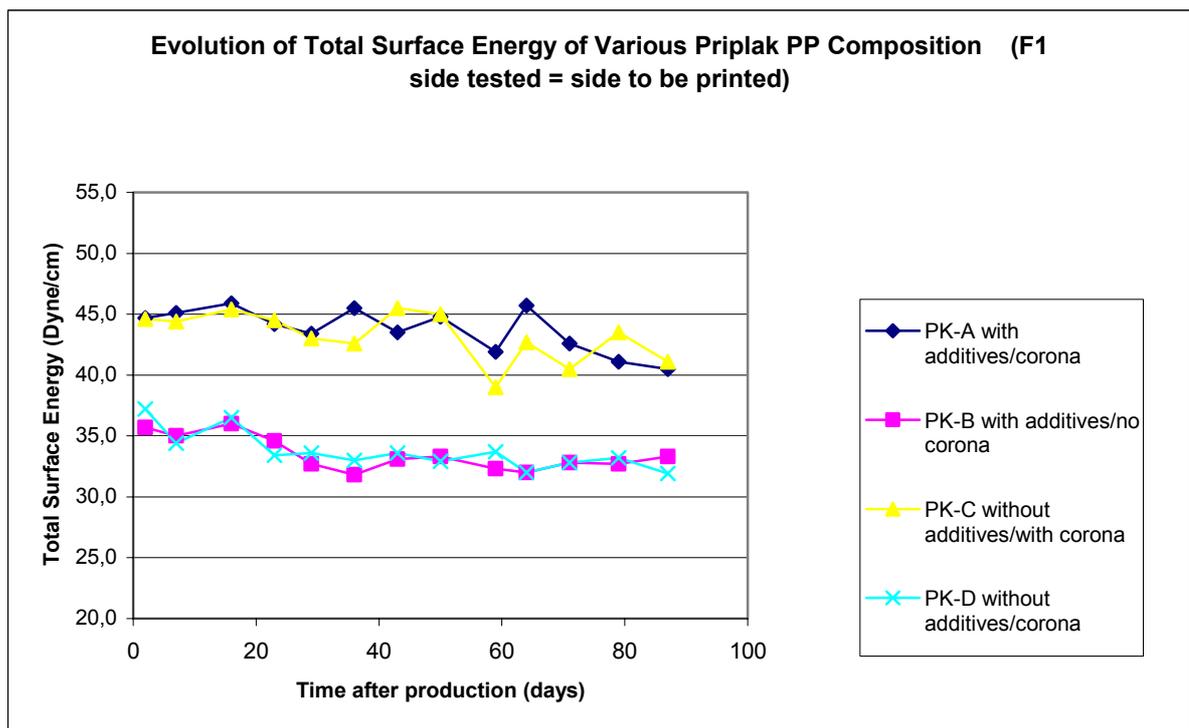
## 6 Investigated surface treatments

In order to evaluate the efficiency of different treatments, they had to be compared to the treatment that currently is done, i.e. corona treatment. Two different PP substrates were used during the project, one substrate with additives (PK-B) and one without additives (PK-D). This was done to see the possible side effect of additives on the surface treatment. The corona treated samples were also compared to untreated PP sheets.

### 6.1 Corona treatment

#### **DAT-test: surface energy**

The surface energy of untreated and treated PP sheets has been measured over a three-month period.



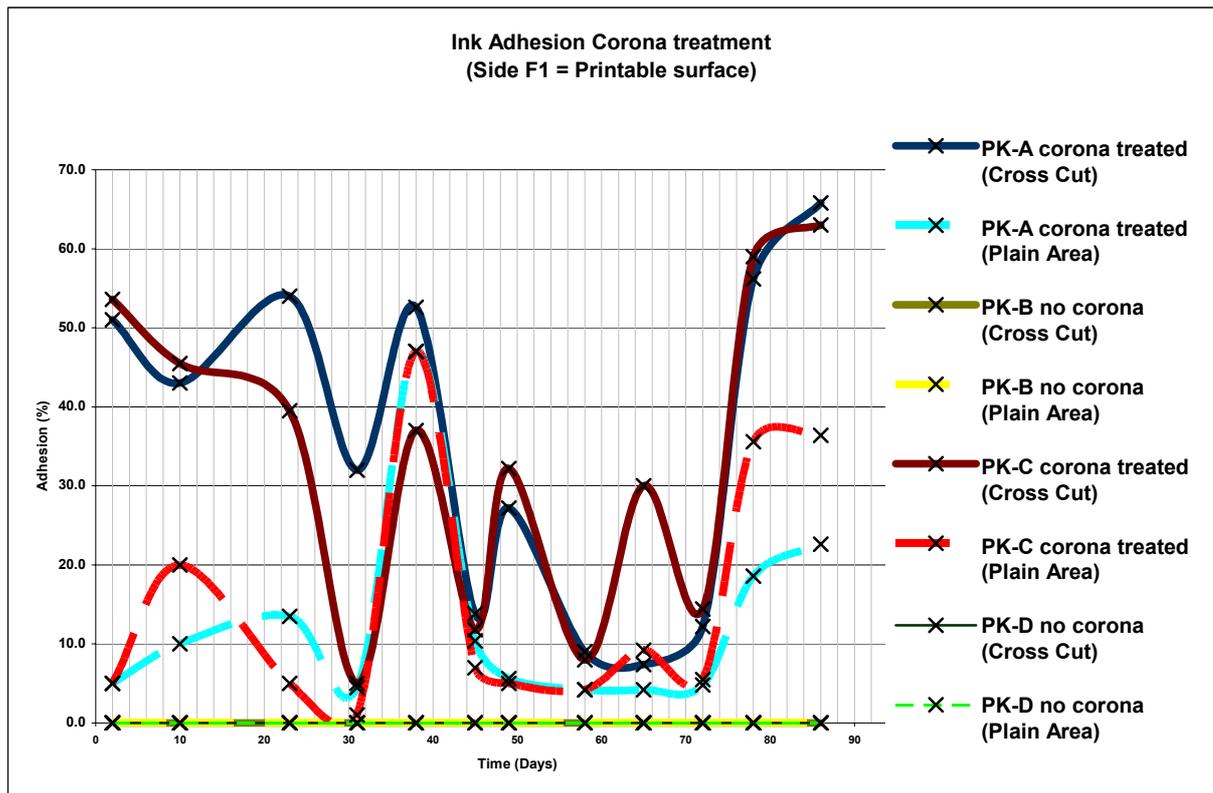
Comments: The substrates with corona treatment have a greater surface energy than the substrates without corona treatment. Within the three months, the surface energy has decreased from 45 dynes/cm to 40 dynes/cm.

This surface property of the material has to be compared to ink adhesion performance over time. Because of the decrease of the surface energy, an

evaluation of other treatments will be done to see if a better result can be received according to time.

### Adhesion test-standard

Ink adhesion was performed on the standard corona treated PP sheets to illustrate the performance of the current corona treatment.



#### Comments:

Adhesion results showed an important variability. This variability is believed to depend on a lot of different parameters, both external parameters (e.g. humidity and temperature – last measurements done on the samples) and test physical parameters (E.g. speed and force).

There is no adhesion at all at the samples that are untreated. The curve in the graph got a trend downwards with time, besides some mysterious high points in the graph. These tops are probably depending at the facts mentioned above. The adhesion decreases from ~50-55% after corona treatment to about 20% after 6 weeks. This can have a connection with the slight loss of surface energy over time on the same substrates and will give us a control to which we will be able to compare the efficiency of other treatments.

## 6.2 Atmospheric plasma

### 6.2.1 Treatment A

This treatment used two different gases (**gas 1** and **gas 2**) to test the effect of various kinds of gases. The surface energy was tested on both sides of the sheets (F1 = side to be printed, F2 = non printable side). \* See appendix A.

### 6.2.2 Treatment B

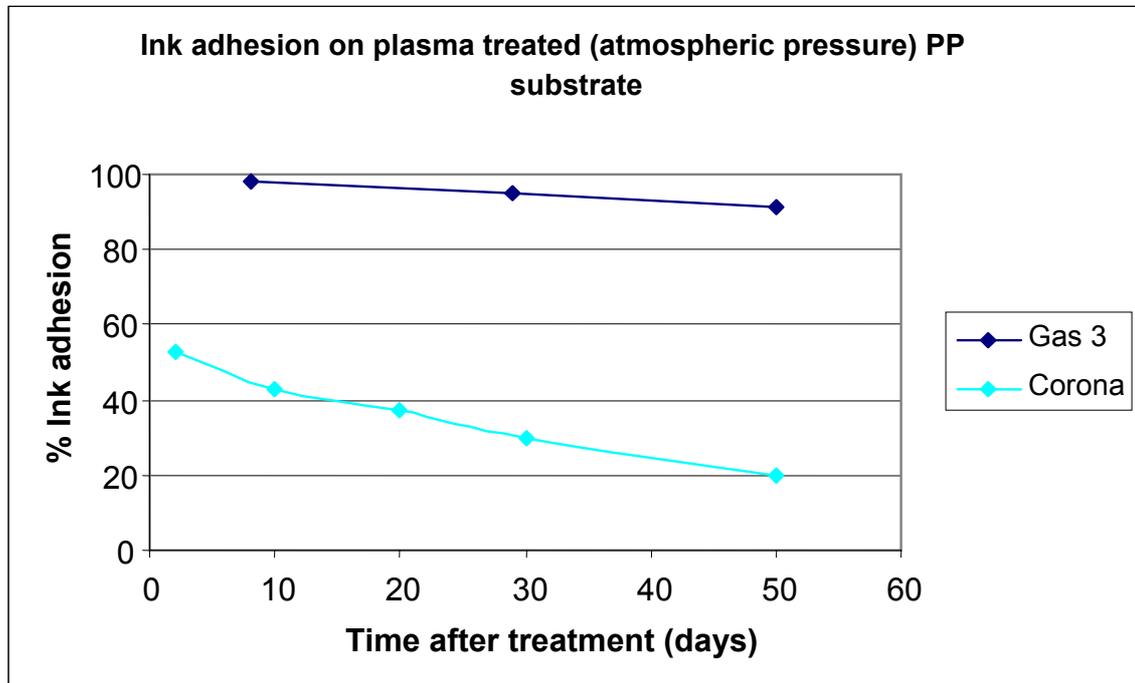
One gas was used in this treatment (**gas 3**). The two different sides of the sheets were tested. (F1 = side to be printed, F2 = non printable side)

<b>DAT-test: surface energy</b>				
Substrate	Side tested	Dispersive (dyne/cm)	Polar (dyne/cm)	Total Surface Energy (dyne/cm)
Test date: 2004-09-06				
PK-B2	(F1)	35.7	7.7	<b>43.4</b>
PK-D1	(F1)	35.7	9.9	<b>45.6</b>
Test date: 2004-17-06				
PK-B3	(F2)	33.6	0.7	<b>34.3</b>
PK-B1	(F1)	35.3	8.3	<b>43.6</b>
PK-D3	(F2)	32.7	0.8	<b>33.5</b>
PK-D2	(F1)	36.0	9.8	<b>45.8</b>

<b>Adhesion test - Standard 15/6</b>				
	PK-B1	PK-B2	PK-B3	<b>Average</b>
cross	100%	99%	98%	<b>99%</b>
plain	99%	97%	98%	<b>98%</b>
	PK-D1	PK-D2	PK-D3	<b>Average</b>
cross	100%	100%	98%	<b>99%</b>
plain	100%	95%	90%	<b>95%</b>

The graph below shows the average ink adhesion performance over time on the PP substrates treated with Gas 3 compared to corona. The level of adhesion is improved compared to corona treatment (~100% compared to

~55% after treatment) and seem to be more stable over time. The level of performance needs to be further followed over a longer period of time.



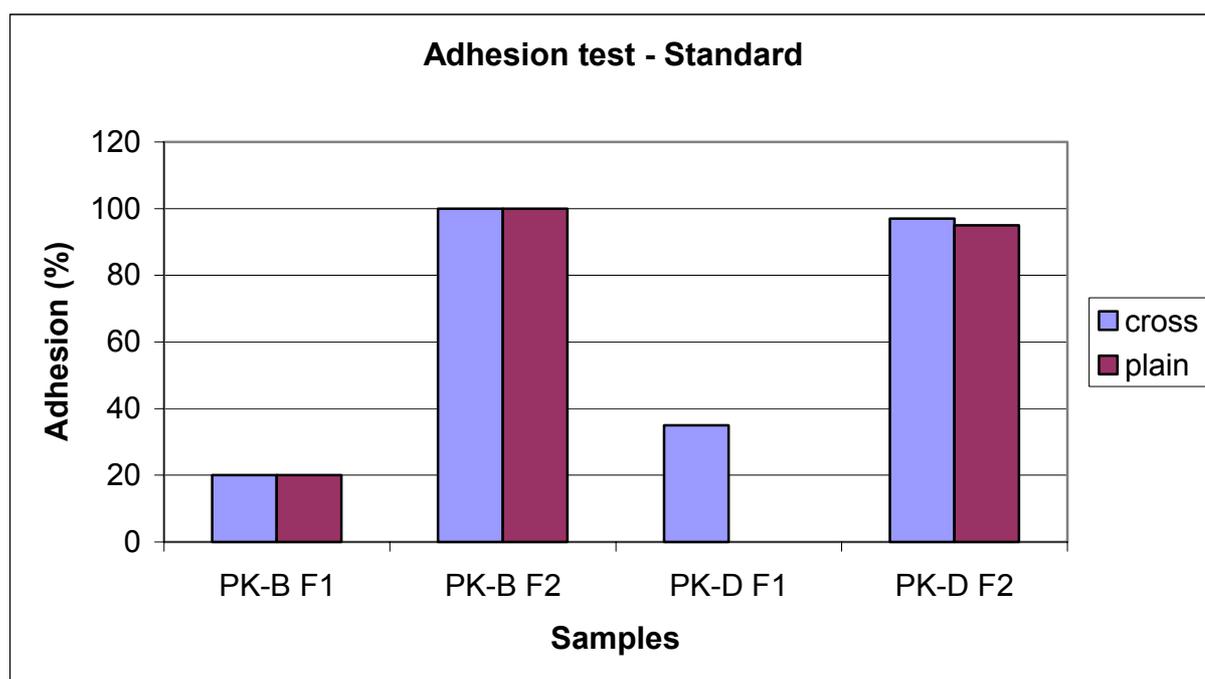
### 6.3 Low pressure plasma

#### 6.3.1 Treatment A

In this treatment only one gas was used (**gas 4**). The two sides of the sheets were tested (F1 = side to be printed, F2 = non printable side). Two different protocols were used on the two substrates provided.

<b>DAT-test: surface energy</b>					
Substrate	Protocol	Side tested	Dispersive (dyne/cm)	Polar (dyne/cm)	Total Surface Energy (dyne/cm)
Test date: 2004-05-21					
PK-B1	1	Treated (F1)	36.6	6.0	<b>42.6</b>
PK-B2	1	Untreated (F2)	37.5	5.3	<b>42.8</b>
PK-B3	2	Treated (F1)	37.0	4.8	<b>41.8</b>
PK-B4	2	Untreated (F2)	36.6	7.4	<b>44.0</b>
PK-D1	1	Treated (F1)	36.1	5.1	<b>41.2</b>
PK-D2	1	Untreated (F2)	35.7	5.4	<b>41.1</b>
PK-D3	2	Treated (F1)	36.9	4.2	<b>41.1</b>
PK-D4	2	Untreated (F2)	37.2	5.3	<b>42.5</b>

<b>Adhesion test - Standard</b>			
	Side tested	cross	plain
PK-B1	Treated (F1)	20	20
PK-B2	Untreated (F2)	100	100
PK-D1	Treated (F1)	35	0
PK-D2	Untreated (F2)	97	95



Comments: The difference of performance which is seen in the results (with same treatment gas) is due to the protocol used.

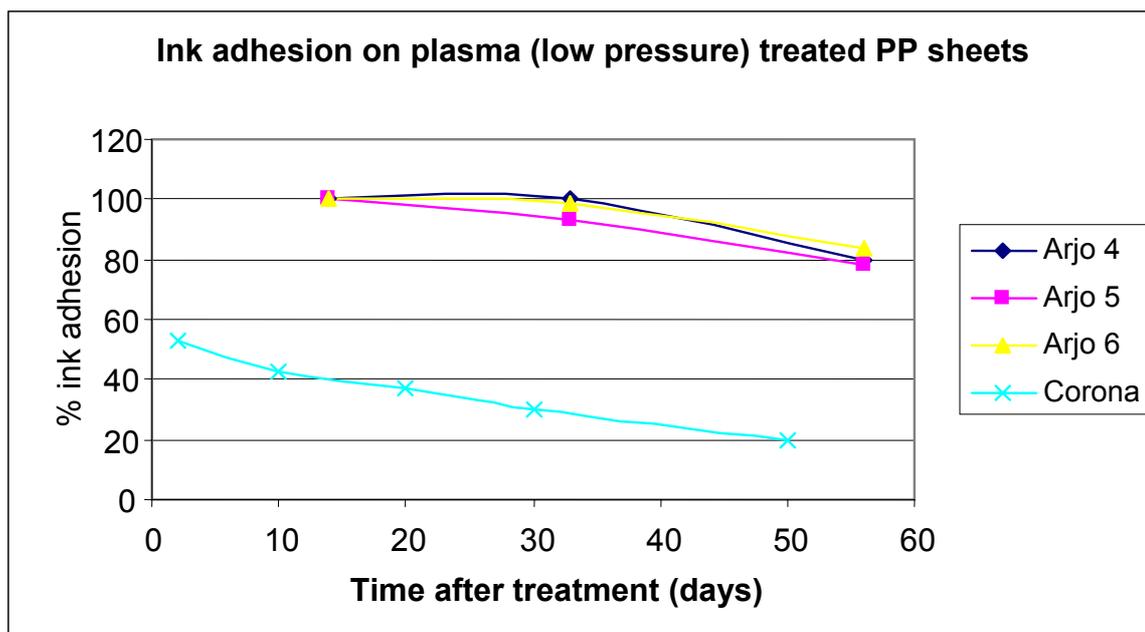
### 6.3.2 Treatment B

In this treatment three different protocols were used (**Arjo 4**, **Arjo 5** and **Arjo 6**). The two sides of the sheets were tested (F1 = side to be printed, F2 = non printable side). \* See appendix B

Very good results from the adhesion test were showed in this kind of treatment:

<b>Adhesion test – Standard</b>		
Samples	Cross	Plain
Arjo4 PK-B1 F1	100%	100%
Arjo4 PK-D1 F1	100%	100%
Arjo5 PK-D2 F1	100%	100%
Arjo5 PK-B2 F1	100%	100%
Arjo6 PK-B3 F1	100%	100%
Arjo6 PK-D3 F1	100%	100%

The graph below shows the average ink adhesion performance over time on the PP substrates treated with the different protocols compared to corona. The level of adhesion is improved compared to corona treatment (~100% compared to ~55% after treatment). Some adhesion is lost after two months but still outperforms the corona. The level of performance needs to be further followed over a longer period of time.



#### 6.4 Plasma grafting

##### 6.4.1 Treatment A

Chemical grafting was used in this treatment. The PP substrate was tested at two sides; inside and outside.

<b>DAT-test: surface energy</b>					
Substrate	Date	Side tested	Dispersive (dyne/cm)	Polar (dyne/cm)	Total Surface Energy (dyne/cm)
XCP	5/5	Outside	32.3	1.1	<b>33.3</b>
XCP	12/5	Outside	35.6	2.6	<b>38.2</b>
XCP	12/5	Outside	32.8	0.5	<b>33.3</b>
XCP	2/6	Outside	36.2	4.7	<b>40.9</b>
XCP	5/5	Inside	36,9	0,8	<b>37,8</b>
XCP	12/5	Inside	34.1	1.2	<b>35.3</b>
XCP	12/5	Inside	32.5	0.4	<b>32.9</b>
XCP	2/6	Inside	30.1	0.3	<b>30.4</b>

<b>Adhesion test-standard</b>			
Substrate	Side tested	cross	plain
XCP	Outside	0	0
XCP	Inside	0	0

This treatment has not been efficient to improving the adhesion performance over corona treatment. Further treatment trials may be required.

## 7 Discussions

Initial improvement on ink adhesion performance and durability over current corona treatment has been obtained with some of the treatments used: atmospheric plasma (treatment B) and low pressure plasma (treatment B).

However, this study has highlighted a strong variability on adhesion results from one sample to the other, within one test, and from one test to the next. After the discovery of these deviations in the standard adhesion test, other alternative methods were tested to measure the adhesion while performing the test under more controlled conditions and test parameters.

### 7.1 Evaluation of alternative adhesion test methods

The standard adhesion test (ASTM D3359) which has been used to test the adhesion between the substrate and the ink at printed plastic samples, has several drawbacks. A lot of individual factors affect the result of the test. For example:

- The temperature / humidity in the room where the test is being done
- The quality of the tape
- The exact angle the tape is being pulled of
- The speed and force the tape is pulled of with
- The carefulness of the applying of the tape (pressure applied on the tape, i.e. level of contact tape/substrate)
- The performance of the cross grid pattern (pressure applied to cut into the substrate affects the level of contact area tape/substrate within the grid)
- The personal estimation of the result of the test

The following tests were assessed as potential equipments to be used as a more reliable adhesion test. The material used in this test evaluation was a corona treated polypropylene (ref. 1106).

### **IGT-test**

The IGT-equipment can be used for different purposes: to simulate offset printing and measure paper/toner adhesion. The standard test method consists of testing the print with pick test oil. The oil used can be of different viscosity (varying degree of tackiness). Therefore a test was being done to discover if this method could be transferred to the plastic materials. The settings for the test at the IGT-machine were 7 m/s<sup>2</sup> constant speed and oils of different viscosity were used to test the adhesion.

Test 1: Medium viscosity oil → 100% adhesion

Test 2: High viscosity oil → 100% adhesion

Test 3: Medium viscosity oil + rougher structure at the wheel applying the oil → 100% adhesion

### **Conclusions:**

The IGT-equipment is not useful for these kinds of tests. The limiting factor in this test was the oil: even the highest viscosity was not tacky enough to give any indication of the true adhesion level of the ink to the substrate.

### **Instron-test**

Test equipment used for testing mechanical properties of papers/plastics called Instron, was investigated for the use of testing ink adhesion onto corona treated Polypropylene. This equipment is also used to test toner adhesion on paper. Therefore similar conditions were used to test the PP substrate. The usual tape (Scotch Magic Tape 810) was used and applied at the surface in the way it is done in the standard test.

Test 1: A speed of 400 mm/min (maximum speed achievable on the equipment) → 100% Adhesion

## Conclusions:

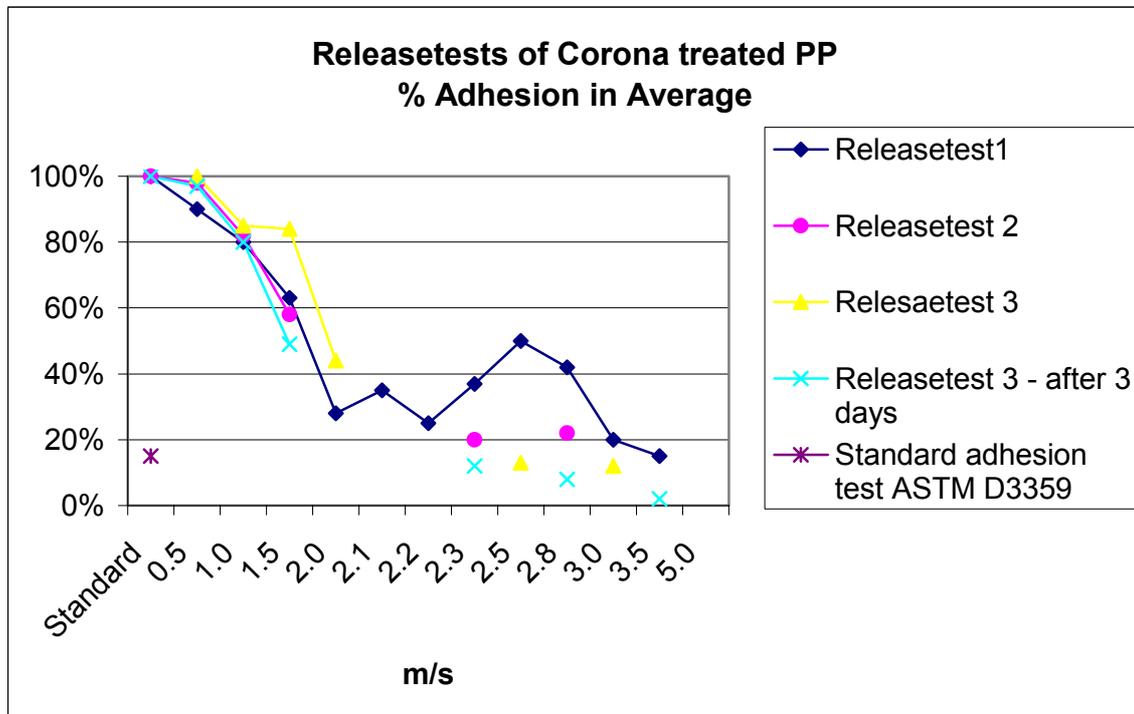
Instron-equipment is not useful for these kinds of tests. The speed at which the tape is pulled is too slow to test the adhesion properties properly at the plastic surface.

## Release-test

This test equipment is used to measure the force required to peel away two surfaces in contact. For the purpose of testing the adhesion at plastic substrates this test turned out to be the most practicable test.

The usual tape (Scotch Magic Tape 810) was used and applied at the surface in the way it is done in the standard test. The sample was fastened in the machine in a proper way and was pulled off according to the settings. The speed could be adjusted from 0.5 m/s to 5 m/s.

Initial results looked encouraging so different speeds and different treated substrates were tested as follows to initiate some statistical evaluation of this technique. Here is the result of the corona treated samples:



There are still some variations of the test results from sample to sample, but results seem to be more reproducible from one series of test to the other (e.g.

results obtained at identical low test speeds). However we can also see that the speed can have an effect on the results; the higher the speed, the lower the adhesion but also the wider spread of results.

The test was also used on plasma treated samples that showed encouraging results:

<b>Release-test Atmospheric plasma (Treatment B) - 18/6</b>			
<b>m/s</b>	<b>PK-B1 Gas1</b>	<b>PK-B2 Gas1</b>	<b>Average</b>
<b>2.0</b>	100%		<b>100%</b>
<b>3.0</b>	100%		<b>100%</b>
<b>5.0</b>	95%	93%	<b>94%</b>
<b>m/s</b>	<b>PK-D1 Gas1</b>	<b>PK-D2 Gas1</b>	<b>Average</b>
<b>2.0</b>	100%		<b>100%</b>
<b>3.0</b>	100%		<b>100%</b>
<b>5.0</b>	95%	90%	<b>93%</b>

Here we can see that the test speed does not have the same effect on the results than the corona treated samples. As the plasma treatment gives higher levels of adhesion to the substrate, high test speeds (greater than 5 m/s) would be required to induce some failure in the test.

The release test came out as the favoured method for testing ink adhesion over the other methods evaluated. It provides controlled test parameters: speed, force and angle of tape pull and can also be fully carried out in a controlled temperature and humidity environment.

However, one of the problems with the release test was that there are no standards available to test adhesion on papers or plastics. So for our purpose, an in-house method has to be developed and compared to the adhesion standard D3359.

The ideal speed for this release test seems to be somewhere between 2-3 m/s. But the research should continue to find out the very best test conditions for this test.

## 8 Conclusions

To print plastic material with a good result is a process that is not easy. One of the main problems is to get the ink to adhere the plastic substrate.

A treatment of the plastic surface has to be done before printing, to avoid delamination of the ink from the substrate.

We have investigated different treatments used on plastic substrates prior to printing. The treatment that seems to give the best result in the tests in this project is the plasma treatment. Some examples of atmospheric and low pressure plasma treatments have shown better results than corona treatment: between 80- 100 % adhesion obtained with all adhesion tests. In the hunt to get a nice plastic printing it is worth to do further research with these treatments.

In the trials with the alternative adhesion test methods the conclusion is that the release test is the most useful in this case. To avoid variability of the results from one test to the other, the external parameters should be more controlled: humidity and the temperature in the room where the tests are performed. We also need to control parameters, like the quality of the tape etc. The effects of these parameters on the test reliability still have to be assessed to draw a final adhesion test method.

However, there will always be some level of variability from sample to sample due to for example non-uniform treatment over a treated area. The corona treatment is also well known for being inhomogeneous, so that can be a factor of variability in the case of these tests.

## 9 Recommendations

Further research and development of an adhesion test method (release test) should be done in the future to confirm if this is a proper method to measure ink adhesion on a plastic material. A wider scale of different substrates and different speeds should be tested to get an accurate result.

## 10 Acknowledgements

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## Appendix A

(1)

DAT-test and adhesion test (standard) of polypropylene treated with; Atmospheric plasma, treatment A

<b>DAT-test: surface energy</b>					
Substrate	Gas	Side tested	Dispersive (dyne/cm)	Polar (dyne/cm)	Total Surface Energy (dyne/cm)
Test date: 2004-06-02					
PK-B	1	Treated (F1)	33.0	7.8	<b>40.8</b>
PK-B	1	Untreated (F2)	36.4	0.9	<b>37.3</b>
PK-B	2	Treated (F1)	34.2	6.7	<b>40.9</b>
PK-B	2	Untreated (F2)	34.4	7.3	<b>41.7</b>
PK-D	1	Treated (F1)	36.6	4.9	<b>41.5</b>
PK-D	1	Untreated (F2)	35.7	1.2	<b>36.9</b>
PK-D	2	Treated (F1)	32.4	1.1	<b>33.5</b>
PK-D	2	Untreated (F2)	36.5	6.8	<b>43.3</b>
Test date: 2004-06-17					
PK-B	2	Treated (F1)	34.3	2.2	<b>36.5</b>
PK-B	2	Untreated (F2)	35.8	5.8	<b>41.6</b>
PK-B	1	Treated (F1)	35.8	5.0	<b>40.8</b>
PK-B	1	Untreated (F2)	32.8	0.9	<b>33.7</b>
PK-D	2	Treated (F1)	33.4	0.8	<b>34.2</b>
PK-D	2	Untreated (F2)	35.7	5.9	<b>41.6</b>
PK-D	1	Treated (F1)	32.8	1.0	<b>33.8</b>
PK-D	1	Untreated (F2)	35.1	5.8	<b>40.9</b>

<b>Adhesion test - Standard 2/6</b>				
cross	PK-D1 Gas1	PK-D2 Gas1	PK-D3 Gas1	PK-D4 Gas1
	2%	2%	100%	0%
	PK-D5 Gas1	PK-D6 Gas1	PK-D7 Gas1	PK-D8 Gas1
	7%	<b>90%</b>	<b>100%</b>	5%
	PK-D9 Gas1			<b>Average</b>
	15%			<b>36%</b>

plain	PK-D1 Gas1	PK-D2 Gas1	PK-D3 Gas1	PK-D4 Gas1
	7%	15%	100%	2%
	PK-D5 Gas1	PK-D6 Gas1	PK-D7 Gas1	PK-D8 Gas1
	5%	<b>98%</b>	<b>100%</b>	3%
	PK-D9 Gas1			<b>Average</b>
	10%			<b>38%</b>
	PK-B1 Gas1	PK-B2 Gas1	PK-B3 Gas1	<b>Average</b>
cross	25%	3%	1%	<b>10%</b>
plain	40%	3%	5%	<b>16%</b>
	PK-D4 Gas 2	PK-D5 Gas 2	PK-D6 Gas 2	<b>Average</b>
cross	15%	1%	12%	<b>9%</b>
plain	2%	2%	15%	<b>6%</b>
	PK-B7 Gas 2	PK-B8 Gas 2	PK-B9 Gas 2	<b>Average</b>
cross	3%	17%	7%	<b>9%</b>
plain	7%	5%	2%	<b>5%</b>

<b>Adhesion test - Standard 18/6</b>				
	PK-D1 Gas1	PK-D2 Gas1	PK-D3 Gas1	<b>Average</b>
cross	0%	3%	5%	<b>3%</b>
plain	0%	5%	2%	<b>2%</b>
	PK-B4 Gas1	PK-B5 Gas1	PK-B6 Gas1	<b>Average</b>
cross	2%	2%	0%	<b>1%</b>
plain	3%	7%	5%	<b>5%</b>
	PK-D7 Gas 2	PK-D8 Gas 2	PK-D9 Gas 2	<b>Average</b>
cross	2%	2%	1%	<b>2%</b>
plain	7%	1%	5%	<b>4%</b>
	PK-B10 Gas 2	PK-B1 Gas 21	PK-B12 Gas 2	<b>Average</b>
cross	7%	7%	5%	<b>6%</b>
plain	10%	15%	5%	<b>10%</b>

Comment: Some initial adhesion results were encouraging on PK-D substrate with gas 1 but further adhesion tests showed inconsistent results.

## Appendix B

(2)

Adhesion test (standard) of polypropylene treated with;  
Low pressure plasma, treatment B

<b>DAT-test: surface energy</b>					
Substrate	Protocol	Side tested	Dispersive (dyne/cm)	Polar (dyne/cm)	Total Surface Energy (dyne/cm)
Test date: 2004-06-09					
PK-B1	Arjo 4	Treated (F1)	28.4	12.5	<b>40.9</b>
PK-B2	Arjo 4	Untreated (F2)	25.0	13.0	<b>38.0</b>
PK-D1	Arjo 4	Treated (F1)	24.8	16.9	<b>41.7</b>
PK-D2	Arjo 4	Untreated (F2)	23.4	16.3	<b>39.7</b>
PK-B3	Arjo 5	Treated (F1)	33.2	3.0	<b>36.2</b>
PK-B4	Arjo 5	Untreated (F2)	30.5	5.2	<b>35.7</b>
PK-D3	Arjo 5	Treated (F1)	31.1	3.8	<b>34.9</b>
PK-D4	Arjo 5	Untreated (F2)	29.9	7.0	<b>36.9</b>
PK-B5	Arjo 6	Treated (F1)	38.4	0.9	<b>39.3</b>
PK-B6	Arjo 6	Untreated (F2)	29.1	11.5	<b>40.6</b>
PK-D5	Arjo 6	Treated (F1)	34.0	2.9	<b>36.9</b>
PK-D6	Arjo 6	Untreated (F2)	34.7	0.3	<b>35.0</b>
Test date: 2004-06-17					
PK-B7	Arjo 4	Treated (F1)	26.2	9.8	<b>36.0</b>
PK-B8	Arjo 4	Untreated (F2)	25.3	17.7	<b>43.0</b>
PK-D7	Arjo 4	Treated (F1)	24.6	16.0	<b>40.6</b>
PK-D8	Arjo 4	Untreated (F2)	23.5	21.5	<b>45.0</b>
PK-B9	Arjo 5	Treated (F1)	33.9	4.3	<b>38.2</b>
PK-B10	Arjo 5	Untreated (F2)	31.4	2.6	<b>34.0</b>
PK-D9	Arjo 5	Treated (F1)	26.6	6.1	<b>32.7</b>
PK-D10	Arjo 5	Untreated (F2)	29.5	7.6	<b>37.1</b>
PK-B11	Arjo 6	Treated (F1)	29.0	3.6	<b>32.6</b>
PK-B12	Arjo 6	Untreated (F2)	24.2	13.9	<b>38.1</b>
PK-D11	Arjo 6	Treated (F1)	24.3	4.7	<b>29.0</b>
PK-D12	Arjo 6	Untreated (F2)	27.1	11.7	<b>38.8</b>

