Revolutionising the future of EVA interlayers: A comprehensive analysis of interlayer properties

Materials Chemistry, Chemistry Department:

Master's thesis

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13.06.2024

Turku

Acknowledgements

I wish to extend my profound appreciation and gratitude to Prof. Carita Kvarnström for her unwavering support, invaluable guidance and feedback throughout the duration of my research and in the preparation of my thesis paper. I would also like to thank Dr. Pia Damlin for her help and knowledge in spectroscopic techniques and characterisation.

I wish to express my gratitude to the University of Turku for the use of their facilities, equipment, and unlimited laboratory time to facilitate the completion of my thesis research. I wish to extend my sincere gratitude to the Materials Chemistry Research Group and its staff, for extending a warm welcome and friendship, and who have aided in my advancement and learning throughout my time here.

This research thesis would not have been possible without the tremendous support of a Cork based manufacturer, Precision Quality Glass Limited and in particular, Dirk and Don. I am deeply indebted for the use of their facilities, materials, manufacturing time, specialist equipment, unlimited access to their laboratory and database resources. I wish to express my deepest gratitude to the entire staff for their hospitality and friendship.

Master's Thesis

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The use of interlayers as performance enhancing materials in the glass industry is standard practice. The introduction of the central layer between glass panes is used to substitute and/or dispense with the need for gases such as argon. Polyvinyl Butyral (PVB), Ethylene Vinyl Acetate (EVA) and SentryGlas Plus/ Sentry Glass Plus Ionoplast (SGP) are the three most common interlayers used worldwide. They each boast numerous advantages such as increased strength, water resistance and good UV resistance, respectively. The most popular type of interlayer used in glass production is PVB. This is due to properties such as transparency, strength, good sound insulation and it is considered cost effective. This research looks at one of the other alternatives, EVA. It investigates the possibility of manufacturing altered EVA to change its properties, with the aim of comparing the enhanced EVA with PVB, EVA and SGP used in the industry on a competitive scale. Numerous tests were carried out to examine the properties of the new EVA samples, these included the crosslinking percentage, the adhesion strength and UV properties. It was found that of the ten samples fabricated for the research, the percentage of crosslinking was high for the majority of samples, with the highest level observed at 93%. The adhesion strength values for most of the samples were above the minimum value, with the highest value for the adhesion strength recorded as 47 N/cm. The UV tests conducted for the transmittance of light indicated favourable results, for three of the samples, with the highest percentage recorded as 99%. The results of these tests were directly compared with previous studies and products manufactured in the industry. The results achieved from performing the tests on the samples yielded a promising outcome for the suitability of possible future use.

Key words: Ethylene Vinyl Acetate; Polyvinyl Butyral; SentryGlas Plus/ Sentry Glass Plus Ionoplast; Crosslinking percentage; Adhesion strength

Abbreviations

ATR-FTIR	Attenuated Total Reflectance Fourier-Transform Infrared
EVA	Ethylene Vinyl Acetate
FTIR	Fourier-Transform Infrared
IR	Infrared
PQG	Precision Quality Glass (company)
PVA	Polyvinyl Alcohol
PVB	Polyvinyl Butyral
SGP	SentryGlas Plus/ Sentry Glass Plus Ionoplast
UV-Vis	Ultraviolet-visible

Contents

1 Introduction

As life progresses in the inevitable advancement of civilisation, so do the fundamental building blocks that humanity have claimed as necessity, for evolution and progression in all societies. The materials and resources we have deemed essential for our survival have progressed and advanced like us, having been adapted to suit our ever-changing needs. This challenge has been accepted in all forms of industry, including the glass sector.

1.1 The history of glass

In our modern age the use of glass has become essential. It is most commonly utilised in the world of construction. It can be seen in almost every building around the world, which is due to one of its most favourable properties, transparency. This allows natural light to pass through, resulting in illumination for longer periods in the day.

1.1.1 The discovery of glass

The origin of man-made glass and glassmaking is still debated among scholars even to this day. A. Berenjian et al. suggests that glass was discovered in the 5th century B.C. by Mesopotamians, while making glazing for cermamics[1]. However, A. Macfarlane et al. suggests that glass first appeared between the period of 3000 to 2000 B.C. with the discovery by the Phoenicians of a clear liquid that appeared with the melting of nitrate blocks used for cooking with sand[2]. Although it appears that the origins may never be known, it is made abundantly clear by the authors that the discovery of glass was plausibly an accidental discovery, similar to many of the world's greatest discoveries.

In ancient times glass was used for a plethora of reasons and this material was not limited to a specific geographical location in the world. Some of the oldest recorded cases of the manufacturing of glass come from Ancient Egypt, Mesopotamia (Northern Syria and Iraq), China and the mediterranean region.

Artifacts recovered support the belief that the Mesopotamians learned how to glaze pieces such as vessels and jewellery with coloured glass[3]. These pieces of jewellery were also very common in Ancient Egypt among the wealthy and the products were fabricated using simple moulds.

Further progression came with the invention of core-forming[3,4]. This is a technique that allows for the manufacturing of vessels such as the examples seen in Figure 2. The object is made by trailing molten glass around a core of a mix of organic materials which include, mud, sand and clay[3]. To decorate the object, the coloured glass is then trailed on top of the surface. The exterior surface is then smoothed by marvelling and the vessel is allowed to anneal for a period of time before the core is removed and the vessel is completed. This process can be seen in Figure 1 below.

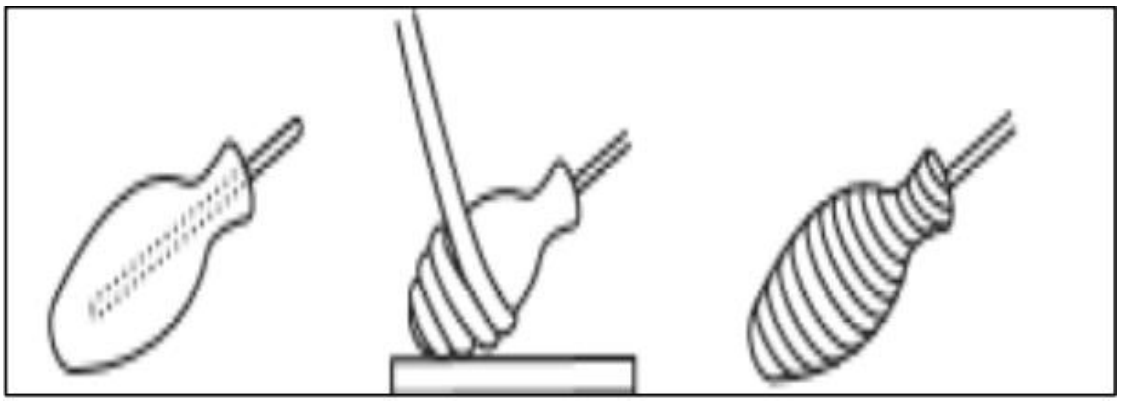
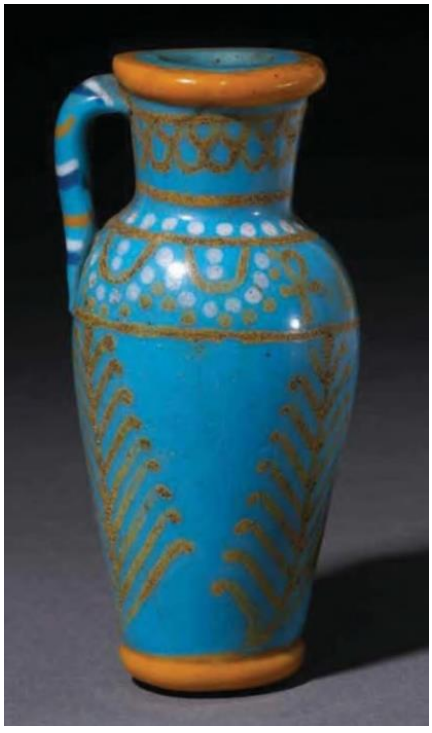


Figure 1. A schematic of the core-forming process[3].

a)



b)

Figure 2. These two images are vessels that were created using core formation and are from the second millennium B.C.[4]

Another technique invented by the Syrians in 300 BC was the craft of glass blowing, which was discovered due to the invention of the blowpipe.[3] This method began to flourish within the Roman empire which allowed for the combination of moulds with this technique, resulting in new shapes and designs such as the vessel seen in figure 3.

Figure 3. The vessel titled 'Lotus Bud Beaker', Roman 1st century C.E., is a product of glass blowing.[3]

1.1.2 The Middle Ages and the development of glass art

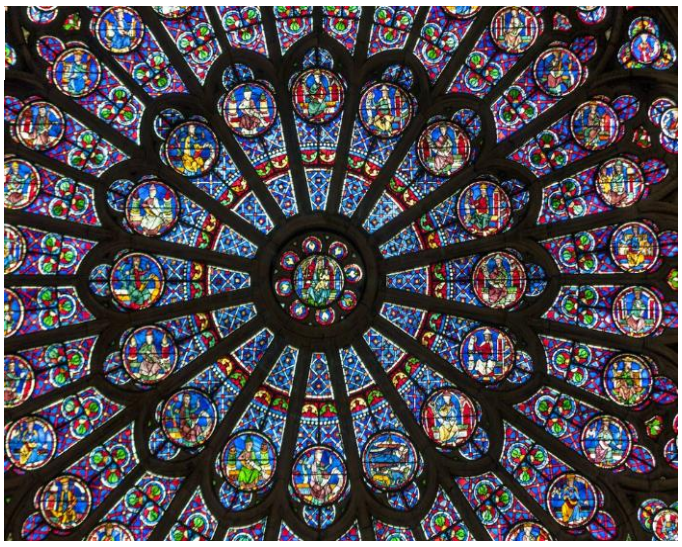
As time progressed, so did the ability to manufacture glass. There is evidence to suggest that the ability to manufacture glass migrated from its original location towards central Europe. This uptake in skill and knowledge was likely due to the expansion of empires such as the Roman empire to other parts of the world, over many years.

M. Vandini et al.[5] sought to compare numerous artefacts recovered from a site in Italy with various pieces collected from other parts of Europe to see if any of the materials were similar in their manufacturing style. It was found that all artefacts tested had similar compositional

features which may indicate "commercial contact" between different established communities, throughout the region. It is however noted that more research must be conducted to establish clear links to further improve the hypothesis suggested.

Glass art which is a display of artistic development is a very common sight in today's society, usually seen in architecture and household items such as glasses and vases, however, the concept of glass art began to thrive during the Middle Ages.

One of the most common types of glass art is stained glass. It is derived from the French word 'vitre' which means window or pane and became popular to produce in the 12th century. Stained glass can usually be found in religious buildings such as churches and is composed of coloured pieces of glass which have been made by painting the glass and fusing it in the oven and mixing the glass with metal oxides[6]. Stained glass was seen as way to express oneself and would often depict events deemed important by those of the time period. The Notre Dame Cathedral in Paris is a prime example of stained glass that is famous throughout the world and an example of a stained glass window from the cathedral can be see in Figure 4 a).

a) 

b) 

Figure 4. a) An example of a stained-glass window from the Notre Dame Cathedral in Paris[7]. b) a modern-day design used in the home as a decorative feature possibly as part of a partial glass door[8].

Venetian glass is another type of glass art that has survived to present day and is still a popular commodity, especially for tourists that visit the area. The secrets of how the glass was produced were guarded and anyone who revealed the information received heavy fines. It is now known that a number of techniques were utilised to create the different products such as drinking

glasses and jewellery. The techniques included, calcedonio and latticinio. The first method simulated marble and other stones and the second method incorporated rods of opaque glass into the glass vessel and this was used to create patterns[9]. As the years went by, the once closely guarded secrets of Venetian glass making were revealed, allowing for others, from different parts of the world, to learn and adopt the trade. Venetian/Murano glass is now readily available to buy in the present day and is seen as an aesthetically pleasing way to create unique and artistic products. Examples of past and present styles of Venetian glass can be seen in Figure 5 a) and 5 b).

a)  **b)** 

Figure 5. a) Replications of 17th century models[10]. b) Current designs available for purchase in Venice[11].

1.1.3 The beginning of glass science

Although glass has been present as a useful material for thousands of years, the concept of "glass science" that has influenced present day glass production, was not invented or discussed until the publication of "Glastechnisches Laboratorium, Schott und Genossen" by Otto Schott and Ernst Abbe in 1886[12,13,14]. Their work looked at the behaviour of glass and the possibility of interpreting the properties of the glass in question. This led others to further test glass properties, allowing for the fabrication of different types of glass samples, with varying structures and characteristics.

Most types of glass up until the 1880's were composed of soda-lime silica and Schott was the first to test and study the effects of adding various substitutions to the samples[12]. Schott and his colleague, Winkelmann, were also the first to attempt to create a model to explain glass

behaviour, by designing a system to calculate the properties of the glass[15,16]. This basis along with the glass catalogue that Schott created allowed for the continuous investigation and understanding of glass materials, which started the revolution of glass development and design.

1.1.4 Glass in modern times

As seen in the previous sections, the concept of glass has been around for many years, with each civilisation improving upon previous ideas. It started out as a material that was sought after for designs and used as decorative pieces for the wealthy classes of the time. However, with the discovery of transparent glass, the concept and use of glass changed with the idea of possibly using it as a functional material, hence the discovery of glass for windowpanes, which is the main use of glass in modern times.

In modern times glass is still utilised in designs and the idea of creating pieces such as vases and jewellery is still prevalent, however, it has moved away from this conceptualisation and glass is a material that can be seen in the majority of current architecture around the world. Glass as a material satisfies a number of requirements that make it ideal as a window feature in buildings. It is transparent, which allows for one to see through it, it is very resistant, and it is able to withstand many different climates and weather conditions. Glass allows the transmission of light and does not deteriorate with the interaction of light, and it is a recyclable material. Whilst the glass manufacturing has a high carbon footprint, the permanency of the material is infinitely recyclable, reusable and refillable, thereby, reducing waste and providing significant savings of our natural resources. Glass is an environmentally friendly material and beneficial to our circular economy, providing employment, thus boosting local economies.

When looking at windows in a building most people are unaware that it is never just a pane of glass facing you. The windows typically will be constructed with two panes consisting of either a gas (argon) or an interlayer between the two panes. The choice of window type will normally be determined by the architect's design proposal for the building.

1.2 The history of interlayers

Approximately, 100 years ago the concept of the interlayer was introduced, this was not to replace the space gap between windowpanes, but to improve properties and the overall performance of the glass product, thereby, removing the necessity for the argon gas. This was achieved through the introduction of an interlayer to create a laminated solution in lieu of the traditional single glass pane to the external window leaf. PVB (Polyvinyl Butyral) was the first interlayer introduced for glass production and today, remains the most commonly used interlayer[17]. It boasts a number of advantageous properties that includes excellent optical properties and large tensile strength. The other main competitor of PVB is EVA (Ethylene Vinyl Acetate). It also has a number of favourable properties which include, excellent optical properties and no delamination. PVB is expensively used over EVA because it is more cost effective to manufacture and offers marginally higher strength properties. An example of an interlayer in glass can be seen in Figure 6 below.

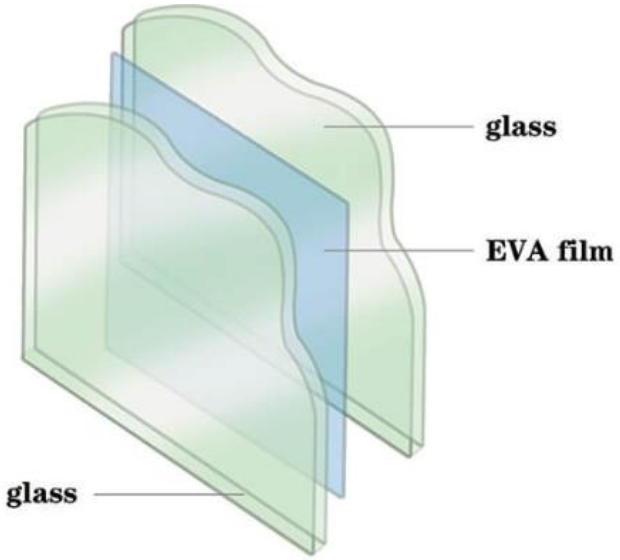


Figure 6. A schematic of an interlayer between two panes of glass[18].

1.3 Polyvinyl Butyral

Polyvinyl butyral (PVB) is defined as a resin that has a number of unique properties such as good mechanical strength, an ability to adhere to glass and an optical clarity that makes PVB an ideal candidate, for an interlayer material, in the glass lamination industry and the structure of PVB can be seen in Figure 7.

The journey of PVB can be traced back to the discovery of polyvinyl alcohol (PVA) in 1924 by Herrmann and Haehnel[19]. This led to the creation and patenting of PVB in 1935. Until this point, the only material used as an interlayer for glass was cellulose nitrate, which was then replaced with PVB as the new front runner in "laminated safety glass for the automobile industry"[19]. PVB was seen as a flexible material that could adapt to the different lamination processes such as vacuum processes with autoclaves, degassing or vacuum calendering.

There are a number of ways to prepare PVB, with each method being unique and usually patented by a specific company that manufactures the material. Time and the invention of new materials and equipment has allowed for more advanced preparation methods with the purpose of altering and customising PVB for specific reasons.

As mentioned previously, it is quite commonly used for automobiles but is also used extensively for buildings as safety glass, due to its ability to "mitigate the hazard from shattered glass fragments" because of its impressive ductility and adhesion properties[20]. It can also be used for photovoltaic cells[21].

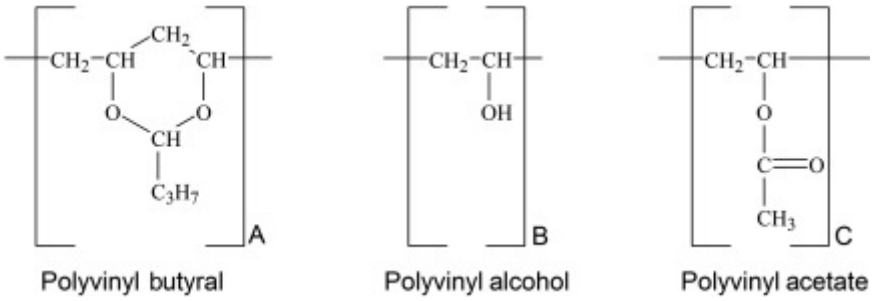


Figure 7. This scheme shows the structure of PVB, PVA and polyvinyl acetate which is one of the starting materials to synthesise PVB[22].

1.4 Ethylene Vinyl Acetate

Ethylene Vinyl Acetate (EVA) is a copolymer which is composed of a strong polar, non-crystalline vinyl acetate monomer and non-polar, crystalline ethylene monomer[23]. As an interlayer it is flexible, strong and does not absorb water, so delamination does not occur. Its chemical structure can be observed in Figure 8.

It was first synthesised in 1928 using a method known as the low-pressure method, however, it wasn't used commercially until the DuPont company filed a patent for it in 1956[24]. Similarly to PVB, there are a number of ways to prepare EVA, with each method being unique and usually patented by a specific company that manufactures the material.

Although it used in the glass manufacturing industry it is also used in other areas, such as. toys, coating and more recently, shoe materials and solar cells.

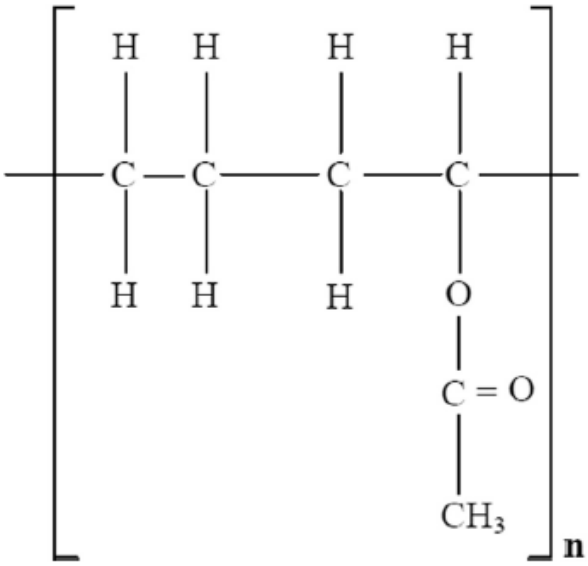


Figure 8. A chemical structure of EVA[25].

1.5 SentryGlas Plus

Sentry Glas Plus or Sentry Glas Plus Ionoplast (SGP) is an ion polymer film. It closely competes with EVA and PVB in the glass lamination market. Its origins begin with a hurricane referred to as Hurricane Andrew that devastated the state of Florida and caused millions of dollars' worth of damage in 1992[26].

Due to the instigation of new performance requirements by the offices of the areas affected, which looked to improve upon the glazing system that was in place for the buildings, a new type of interlayer was created. Of the three interlayers discussed in this research, SGP was the newest interlayer to hit the market created by the DuPont company is 1998[27]. The aim of creating this new type of interlayer material was to try to enhance and improve some of properties already exhibited by both PVB and EVA such as the strength, adhesion and clarity of the product.

When compared with PVB and EVA, SGP is shown to have a good durability and is less sensitive to moisture absorption and is comparably better than PVB in this regard, however, EVA is still a better candidate if water absorption and delamination is being considered. It has excellent mechanical properties and a high strength and if compared with PVB at the same thickness, SGP has twice the interlayer bearing capacity under the same load[28].

1.6 The use of interlayers in the glass industry

Interlayers such as PVB, EVA and SGP are used in the glass industry due to their unique properties which make them ideal candidates. The most common use for interlayers and glass is as windows. Many buildings will have interlayer windows instead of windows with gas in between the panes. This will normally be determined by the engineers and architects building the structures, where certain aspects will be taken into account. For example, in very damp climates water may cause delamination, therefore, EVA may be considered. The strength and durability of the product will also be considered when building, so PVB or SGP may be considered as alternatives. Due to the flexibility of the interlayers, they would also be ideal candidates for buildings that are being designed to have a unique structure and are also very popular for interior design projects such as glass floors (see Figure 9 a-c).

a)

b)

c)

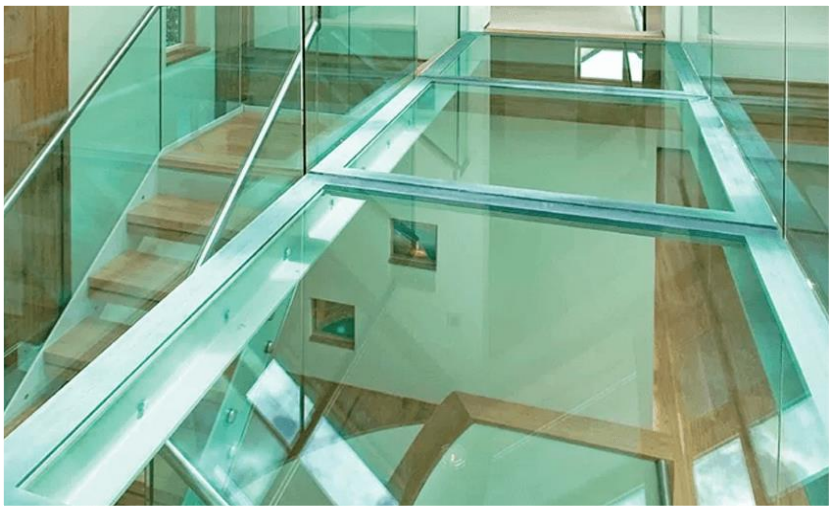


Figure 9. a) The EVA interlayer is often used in windows in ships because delamination will not occur with this material[29]. b) All three interlayers are flexible and will often be used when building unique structures[30]. c) Interlayers are often used in glass for interior design such as for floors and glass barrier for balconies due to their strength[31].

1.7 Aims and Objectives

The aim of this research is to fabricate a number of EVA interlayers and to assess their properties. This will be achieved by working closely with Precision Quality Glass Ltd and the University of Turku. Each interlayer will be manufactured with the objective of tailoring each test sample to attempt to improve or enhance a specific property that EVA interlayers exhibit. These properties include, good adhesion, a high crosslinking percentage and good transmittance. Tests to assess these properties will be conducted in laboratories in both Precision Quality Glass and the University of Turku. All results will be compared with industry level standards.

2 Experimental Work

2.1 Materials

All materials used in the research were provided by Precision Quality Glass Ltd. Ethylene Vinyl Acetate (EVA) foil, Xylene, Deionised water (H_2O), Methyl ethyl ketone (C_4H_8O), float glass, release film and ESA HA-P 24/36/48-66 permeable tape (laminating tape). Due to the nature of the research conducted the products that were added to the main component to create the foils may not be disclosed, so the names that they are to be referred to for the purpose of this paper, are products 1-10.

2.2 Fabrication of EVA interlayers

For this research, 10 interlayers were produced, and each interlayer followed the same experimental method that will be described in detail in this section. Each interlayer had a different and specific product added to the master batch, to alter the original EVA foil, with the intention of testing the new foils for direct comparison with previous studies and other interlayer products available, with the aim of improving targeted properties of EVA. As mentioned above in section 2.1 Materials, the products added must be referred to as products 1-10 in relation to foils 1-10.

Firstly, 100g of EVA foil was weighed using an electronic balance (Figure 10 a)) and then cut into small pieces. The EVA was placed in a beaker and placed to the side. Secondly, 200ml xylene was poured into a beaker with a lid and was placed in an oil bath that was on a hot plate. A stirrer and a thermometer were inserted into the liquid through the top of the lid and the hot plate was turned on. The stirrer was rotating at 200 rpm throughout the process. Once the xylene reached a temperature of 35℃, the cut-up EVA was placed piece by piece into the heated xylene. The next step was to add the product associated with the foil, e.g., if foil 4 was being produced, product 4 would be added as the next step.

The mixture which is known as the master batch in this field of study, was allowed to reach a temperature of ~ 85℃. It is important to note that the temperature could not rise above 90℃, as this is when crosslinking begins to occur and would affect the final interlayer. As the temperature was kept at 85℃ the EVA and the product mixed with xylene and eventually resulted in a master batch with a honey-like viscosity (Figure 10 b)). This process took approximately two and half hours each time for every foil fabricated.

The final step in the process was to place a large pane of glass measuring 1m × 1m on a flat surface and tape the edges of the pane using electrical tape. This was done to stop the master batch from overflowing. After stopping the stirrer, the beaker was taken immediately from the hot plate and the master batch was poured carefully onto the pane whilst a second person used a straight edge to spread the master batch evenly and smoothly over the entire glass pane (Figure 10 c)). It is important to pour the master batch right away as the xylene begins to evaporate immediately at room temperature and it is also essential not to remove the straight edge until the spreading is complete to avoid bubbles and different thicknesses forming within the interlayer.

The foil was then left to dry for 2-3 hours to allow the xylene to evaporate and was rotated frequently. It was finally placed on a drying rack for 4 days to ensure that all of the xylene had completely evaporated from the foil (Figure 10 d)). Each foil was prepared in this way, with a total of 10 foils manufactured.

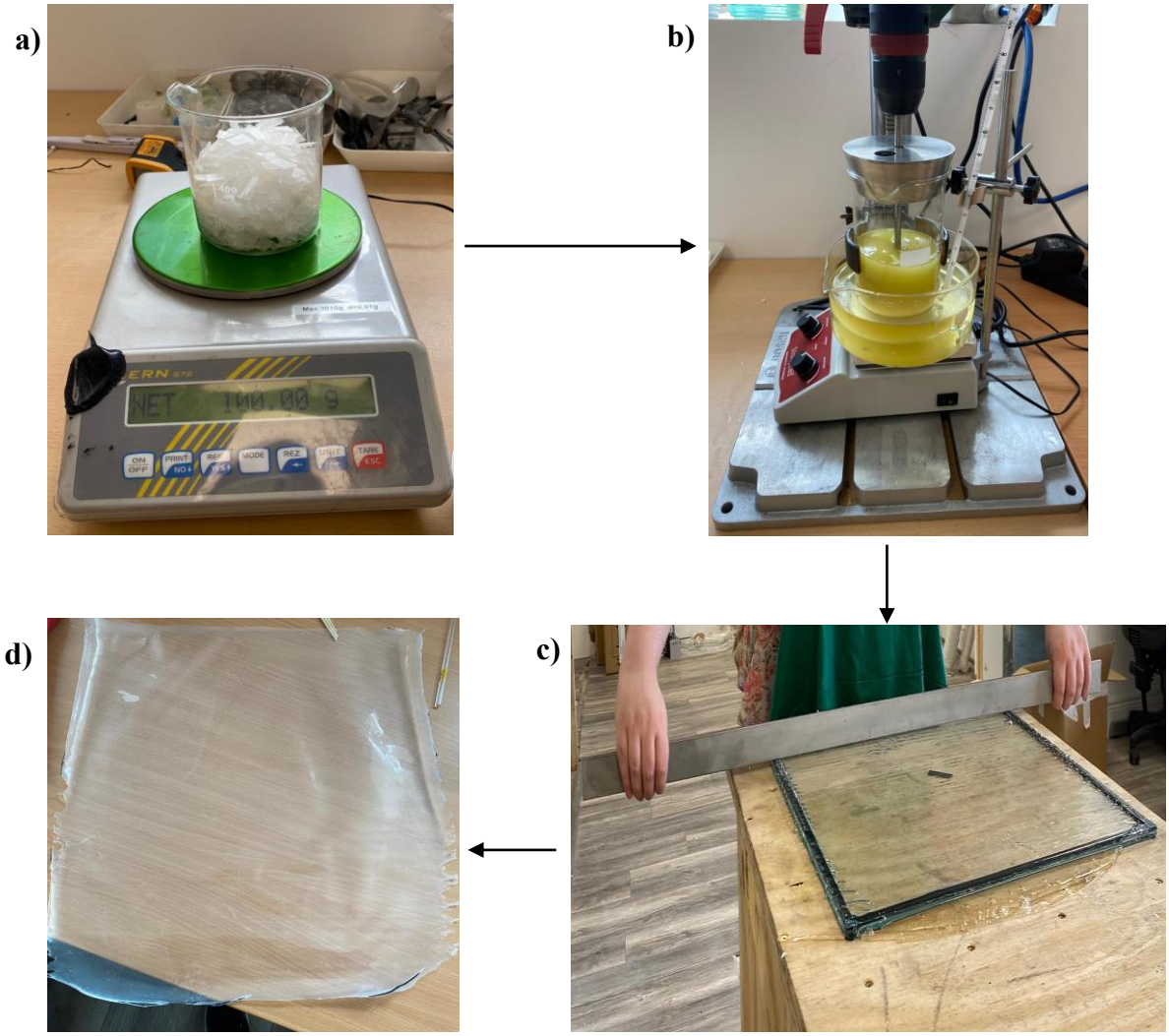


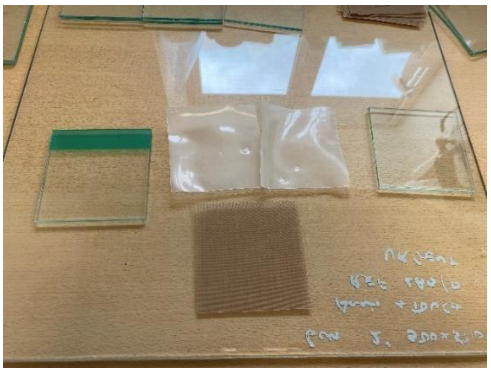
Figure 10. a) This is the first step in the manufacturing of the interlayer, EVA is weighed out accurately using an electronic balance. b) This shows the master batch being mixed. c) The master batch has been poured and is being spread evenly on the glass pane. d) The interlayer ready for the next step in the process.

2.3 The assembly of the glass samples for lamination

For testing purposes two glass samples for each foil totalling 20 samples were prepared and assembled as seen below in Figure 11. The first glass sample in each case was used for UV testing and the second sample was used for adhesion strength testing and the gel rate value. It is important to note that for the samples to be accurate for measurements, the thickness of the foil for each sample must be between 1.6-2 mm. Therefore, each of the 10 foils used were measured using a vernier callipers and each thickness was recorded.

Each UV sample was prepared the same way for each foil as was each adhesion testing sample. The UV samples were created by firstly taking a square of glass and cutting out an equivalent square of foil. Secondly, two glass squares were cleaned to ensure that there was no contamination between the glass and the foil which would affect the results of the future tests. The foil was then placed on one of the squares of glass, while the second square of glass was used to close (sandwich) the foil and the sample was held together using green lamination tape, ensuring all four sides were covered. This tape was not only used to keep the sample together, but also to stop the foil from spilling out once it was placed in the laminating oven. This process was repeated for all 10 foils.

The samples for the adhesion testing were prepared in a similar manner, however before placing the foil on the cleaned glass square, a strip of green laminating tape was placed on top of the glass. The sample was then formed in the following way, the foil was placed on the glass with the green laminating tape and then a release film was placed on top of the foil (this was also the same size as the glass and foil). Finally, the second square of glass was placed on the release film and the sample was held together with green laminating tape. The release film was used so that the second square of glass could be removed after the lamination process, allowing for the exposure of the foil for testing purposes (Figure 11).

a) 

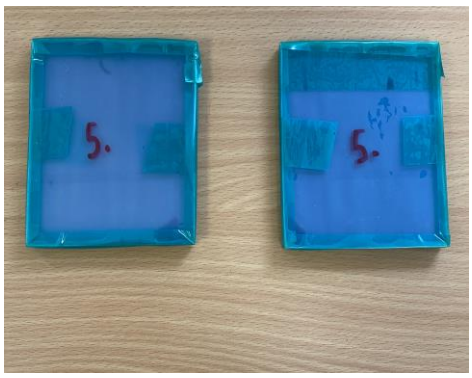
b) 

Figure11. a) The materials for the assembly of the adhesion sample b) The two test samples ready for the laminating oven.

2.4 The lamination process

The twenty prepared samples were taken and placed on a red canopy on a large tray that is pushed into the laminating oven. The samples were spread out and placed between aluminium bars that are visible underneath the tarp of the tray (Figure 12 a)). The rest of the red canopy was placed over the samples and a mesh was placed on top of this (Figure 12 b)). Finally, a blue mat was placed over everything and was secured to ensure that the samples didn't move

during the laminating process (Figure 12 c)). All of the mats are needed between the blue mat to guarantee that the samples do not stick together when the vacuum is on in the laminating oven. The tray was then pushed into the oven and the laminating process began (Figure 12 d)).

a)

b)

c)

d)

Figure 12. The four steps to setting up the samples for the laminating process.

The laminating of the foils was a three-step process as seen in Figure 13 below with SP01, SP02 and SP03 referring to the temperature at each step and MN01, MN02 and MN03 referring to the amount of time each step takes in the process. The oven used was a calibrated oven. Before the samples were heated the vacuum pump was used to remove any air present between the glass and the films. Once this was achieved, the oven was turned up to 60℃ and the films began to melt. This is known as the melting phase, and it lasted for 1 hour and 6 minutes. Once this cycle was complete the second step began. The oven was turned up to 125℃ and cross-

linking occurred in the foils. This allowed the foils to stick to the glass. This step was known as the curing phase, and it lasted for 1 hour and 5 minutes. The final step keeps the temperature at 125℃ for 1 minute and then the process is complete. It is important to note that the pressure was kept at 1000 mbar for the three-step process. The samples were then gradually cooled down and once ready the vacuum pump was turned off and the test samples were ready for testing.

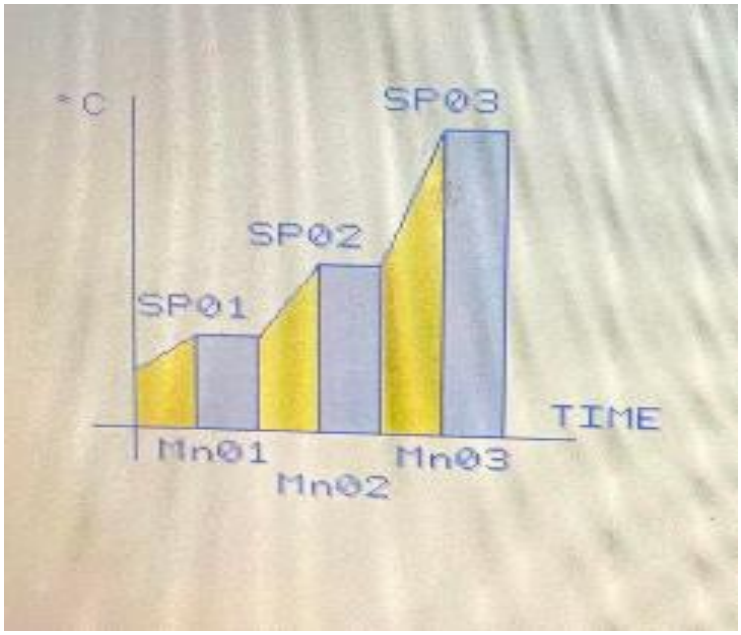


Figure 13. A graph of the three stages of the lamination process.

2.5 Materials Characterisation

A number of tests were performed on the samples to allow for a comparison of results between the foils and products already in use.

2.5.1 Peel tests for adhesive strength

The peel test checks the adhesive strength of the foil to the glass. In this case adhesion refers to how well the foil will stick to a pane of glass and whether it can be pulled away easily or not at all (see figure 14). There are two types of peel tests that can be used to assess the adhesive strength of the foil, and these are referred to as a 90° peel test or a 180° peel test[32]. The names refer to what angle the foil will be peeled at. The foil is always tested at room temperature and thickness of each foil should be within the same range for the parameters of the testing. The type of machine used will determine how fast the foil can be pulled away. With certain types of machines different speeds can be applied to see if it may affect the overall results. This is also the same for the angle. Certain machines can only test the foil at one angle whereas other machine may have adjustable arms that can test at more than one angle. Both types of angle testing are extremely accurate, however, a 90° degree peel test can perform measurements over a much broader range for adhesive samples than a 180° degree peel test. In the work performed for this research a 90° peel test was used.

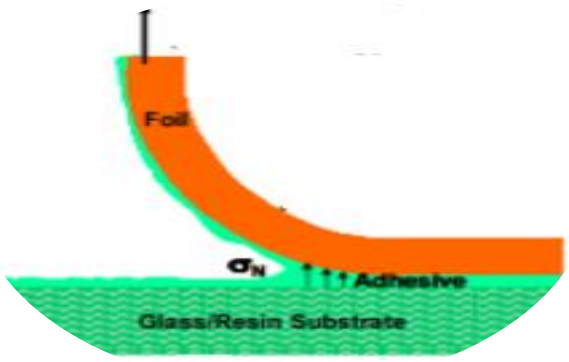


Figure 14. An image explaining the concept of adhesion in the context of glass manufacturing[33].

Firstly, the tape was removed from each of the samples. Half of the samples were being used for this test and the other half were placed to the side for testing later. Any excess foil that had expanded in the oven was removed from the sides of the glass. The top piece of glass was removed using the release film, leaving the foil exposed for testing. Each sample looked identical with a layer of glass, a layer of foil and a strip of the green laminating tape on top. Using a ruler the foil was cut into four strips for peeling. Four strips are normally used to see if the adhesion is the same throughout the foil and an average value is normally calculated using two of the strips. Each strip must be identical, because 1mm more or less will result in

10% less adhesion and will interfere with the accuracy of the results. It was also essential that the edges on each side of the sample were not used as the outer edges of the foil react with oxygen making it unable to use these pieces for testing (Figure 15).

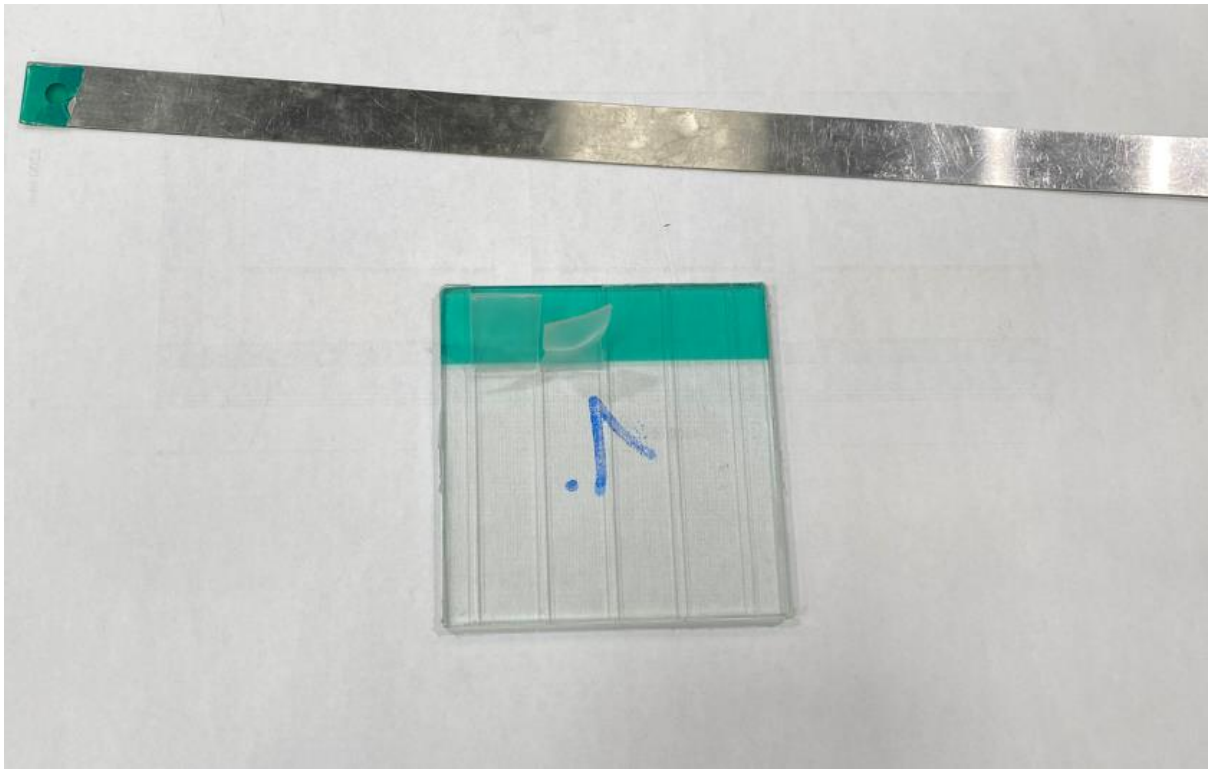


Figure 15. The foil cut into strips and ready for testing.

Once the sample is ready for testing, the machine (force gauge) for the peel test is set up and the first strip is attached to the machine as seen in figure 16. The machine is turned on and it begins to peel the strip back until it reaches a point where it can no longer do so. At this point it measures the force needed to peel the foil and the value is recorded. This is repeated for each sample and all values are noted. In all calculations the friction between the machine and the surface it is placed on is taken into account and it factors into the calculations to determine the adhesive strength of each foil.

Figure 16. Images of the force gauge.

2.5.2 Testing of the crosslinking rate (gel rate value)

The strips that were used to test for the adhesive strength were retained and used for the next test which was the crosslinking rate. Crosslinking is defined as a short sequence or a bond that will link polymer chains together and it is important because it will improve the stability and the elasticity of the material.

1g of the first foil was cut up and weighed. This value is equivalent to 100%. The value will be re-weighed after the process and the new value will tell us how much of the material has cross-linked. The foil was placed in an egg cup, and this is placed in the Soxhlet extractor. The reaction was started and was a reflux reaction. The reaction used xylene to wash the foil and took approximately four hours to complete. Once it was finished the pieces of foil were removed and placed on a petri dish to dry as seen in figure 17. This allowed any xylene in the pieces to evaporate. Once the pieces were dry, they were re-weighed, and the new value represented how much of the foil had crosslinked and the difference between the original and new value was how much of the material had been washed away by the reaction due to not crosslinking. This process was repeated for each foil and the crosslinking rate was calculated for each sample.

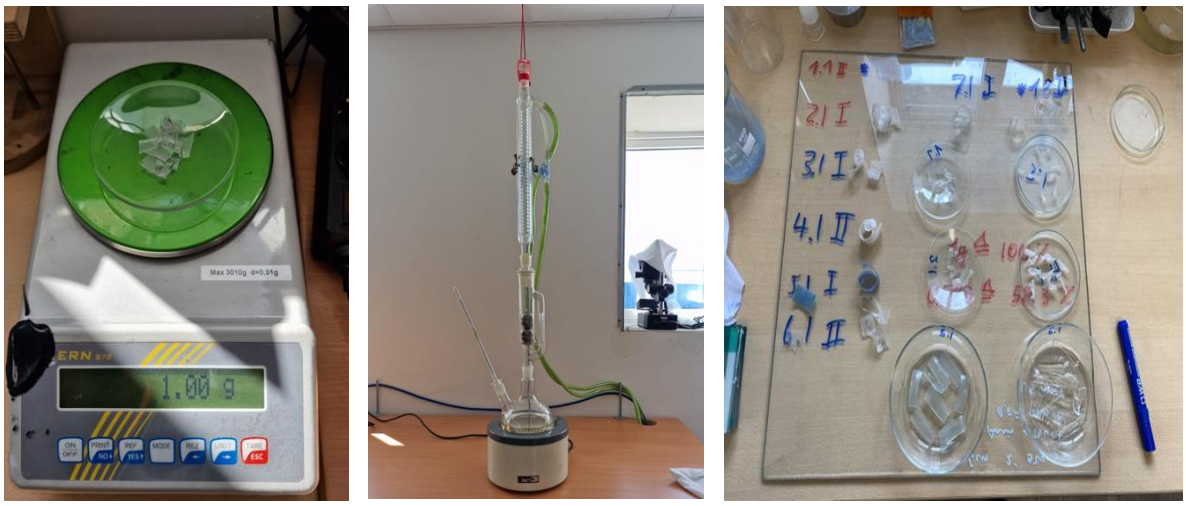


Figure 17. The foil is weighed, washed and then re-weighed to determine the crosslinking rate.

2.5.3 Optical measurements

The second set of samples that were prepared and laminated were now used for the optical measurement tests. Using a UV-Visible spectrophotometer by Labnics, each sample was placed in the machine within a holder to keep the samples in place. The spectrophotometer was turned on and the absorbance of each sample was measured at a specific wavelength using a transmission mode. Each value was noted and using the measurements the transmittance for each glass sample was calculated.

Figure 18. A UV-Vis spectrophotometer.

2.5.4 Fourier-transform infrared spectroscopy (FTIR)

Using a Fourier transform infrared spectroscopy machine by Bruker, ATR-FTIR spectroscopy was used to produce a spectrum that would help in the analysis and identification of functional groups in each sample. ATR is short for attenuated total reflectance and is classified as a sampling technique that is combined with infrared spectroscopy to examine and analyse liquids and solids. It is a popular technique as there is no need for sample preparation. The sample for testing is placed in the machine on top of a crystal (in the case of this research a diamond) and it is held in place. Using total internal reflection an IR light is passed through the diamond which results in the light reflecting off the surface that is in contact with the sample, and evanescent waves are formed which move through the sample. The beam is collected by a detector as it leaves the crystal. This allows for the collection of the data and the production of spectrum to be analysed.

A strip of each foil that had previously been tested for the adhesive strength was used in each case and a number of spectra for each foil was recorded. A comparison of the foils before lamination, after lamination and after the crosslinking rate test were sampled, with the aim of comparing all spectra with their counterparts, to understand if there were any fundamental differences present.

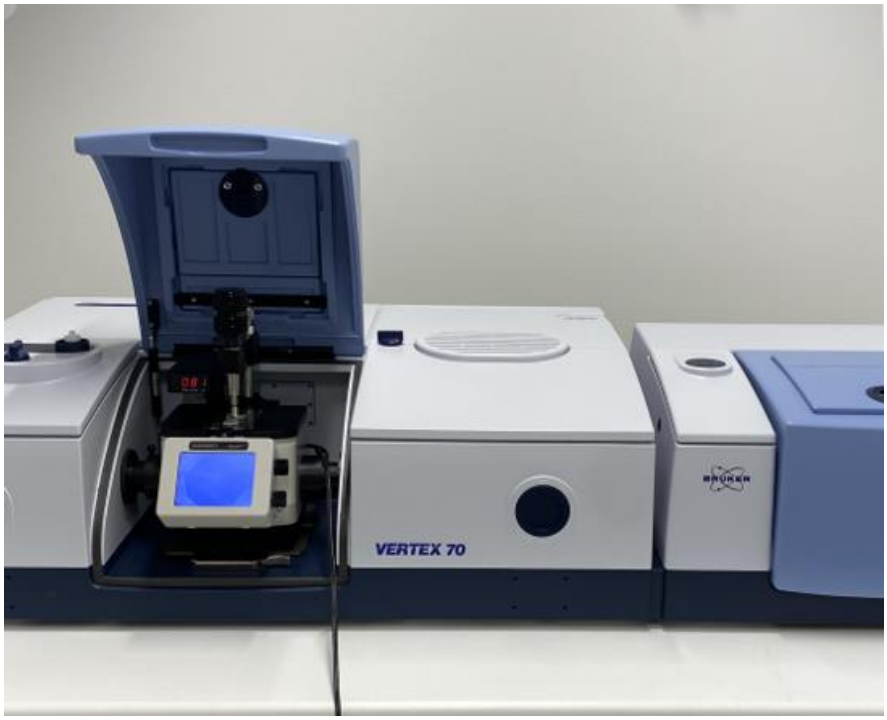


Figure 19. An FTIR spectroscopy machine.

3 Results

3.1 Materials characterisation

Originally seven foils were fabricated for testing purposes, and these refer to foil 1-7 in the tables below. However, after the testing had been completed for each foil, it was noted that foil 7 showed impressive results in the majority of tests. It showed good adhesion, transmission and a good crosslinking percentage. Therefore, it was decided that foil 7 would be re-made to see if the results could be replicated. To understand how the additive had affected the results, foil 7 was created the same way as in the previous attempt, however, different concentrations of the additive used were added to see if the results would change. This is why foil 7A, 7B and 7C appear in the table. One is a replication of 7 and the other two are the foils with different concentrations added. The original foil 7 had a concentration of 1ml, foil 7A had 0.5ml added to it and foil 7C had 0.25ml added to it, with foil 7B represented the duplicate of foil 7.

Foil 1 and foil 1R are the reference foils and represent the standards sought after in an industrial setting and respective of the company foils used in manufacturing. This foil is roughly 0.3mm in thickness and before lamination is opaque and white. It has a rough surface and can be hard to cut through when using it for testing purposes. When compared to the other foils it can be seen that it is not as smooth as the other test samples prepared. After the lamination process it becomes completely transparent and it has excellent adhesion to the surface of the glass.

Foil	Foil Thickness (mm)	Adhesive Strength (N/cm)
1	0.22	40
2	0.18	37
3	0.24	29
4	0.2	33
5	0.25	17
6	0.21	32
7	0.19	43
7A	0.16	39
7B	0.22	0
7C	0.3	47
1R	0.23	0

Table 1: A table showing the results recorded for the adhesive strength for each foil.

This first table looks at the adhesive strength results for each foil tested. The minimum number expected in the industry is 20 N/cm, however, the higher the value the better the foil as this is indicative of a very good adhesive strength. The reference foil 1 showed a value of 40 N/cm and it was found that foil 7 was able to exceed this value, showcasing a promising result. It was found that foil 7C showed an even higher value of 47 N/cm. A higher value than 7 was expected due to a higher concentration of the additive that was used for the preparation of foil 7 being added. Foil 5 showed a low value at 17 N/cm; however, this was also anticipated because this foil was a thermochromic foil and these types of foils will usually have a lower adhesion to glass than standard foils for glass. Unfortunately, both foil 7B and 1R could not be tested and they yielded values of 0. It is speculated that the foils had been contaminated during the fabrication process. This means mean values of the foils could not be represented on the table either due to the 0 values for the replications. Due to a specific time frame these foils could not

be replicated and re-tested to determine accurate values and had to be disregarded due to the inability to compare them with other foils.

Foil	Foil Thickness (mm)	Crosslinking Percentage (%)
1	0.22	89
2	0.18	89
3	0.24	91
4	0.2	78
5	0.25	92
6	0.21	74
7	0.19	91
7A	0.16	92
7B	0.22	0
7C	0.3	93
1R	0.23	0

Table 2. A table showing the results recorded for the crosslinking percentage/rate for each foil.

Table 2 looks at the results from the crosslinking rate. A crosslinking rate of 70% is the minimum value one looks for, when performing this test, however, like the adhesive strength, the greater the value the better the foil. Similarly to table 1, no values were recorded for foil 7B and 1R, so foil 1 acts as the only reference foil. Foil 1 showed a value of 89% with this being matched or exceeded by 6 of the other foils. Surprisingly foil 6 had quite a low value at 74% and foil 5 the thermochromic foil had quite a high value at 92%. To try and understand these values, the foils would need to be re-made and the crosslinking rate re-done in the future.

3.2 The UV-Vis performances

Using a UV-Vis spectrophotometer the absorbance for each sample was recorded at a wavelength of 500nm and can be seen in table 3 below. These values were then used to calculate the values for the transmittance.

Foil	Foil Thickness (mm)	Absorbance (a.u.)	Transmittance (%)
1	8.220	0.012	97
2	8.180	0.233	58
3	8.240	0.751	18
4	8.200	3.254	0.06
5	8.250	2.313	0.5
6	8.210	0.006	99
7	8.190	0.015	97
7A	8.160	0.149	71
7B	8.220	0.400	40
7C	8.300	0.010	98
1R	8.230	0.283	52

Table 3. A table showing the results recorded for the absorbance and transmittance for each foil.

Using the values for the absorbance the transmittance values for each sample were calculated and recorded in table 3. Foil 1 was used as the reference for these values, and it had a transmittance of 97%. This is in line with the value that is expected with glass products in the industry and is usually the expected value that should be reached for glass window products that are being released into the marketplace. The transmittance values for foils 3, 4 and 5 were exceptionally low, with limited transmittance. These values were expected based upon visual

examination of the samples before testing. This indicates that foil 3 and 4 would be unsuitable to use for glass windows and other glass products that require transparency. However, the intent of foil 5 was not to make it a transparent product as it is a colour changing foil, so the results for the transmittance are not unfavourable. Foil 7 and 7C showed great results with values of 97% and 98% respectively, however, foil 6 showed the best result overall with a very high value of 99%, which is an excellent result.

3.3 Fourier-transform infrared spectroscopy (FTIR) results

Due to the nature of the research an in-depth analysis of the foils cannot be discussed in this paper, therefore in this section the commentary and observations in regard to the FTIR spectra may be limited.

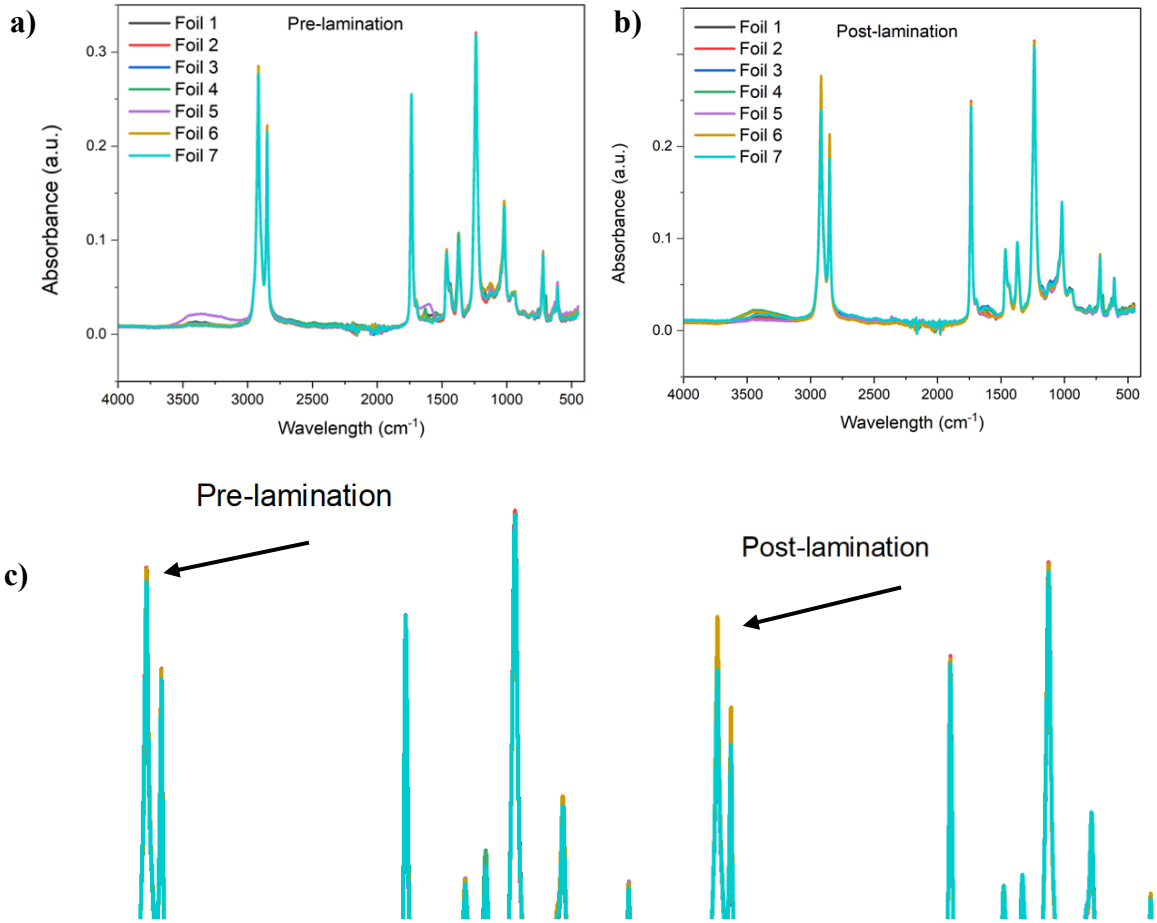


Figure 20. a) A graph of the first 7 foils before lamination. b) A graph of the first 7 foils after lamination. c) This shows the difference in intensity between foil 6 before and after lamination.

ATR-FTIR with a resolution of 4 and a scan rate of 32 was performed on the strips used in the adhesive strength test and a number of spectra were recorded. The graphs in figure 20 are of the first 7 foils manufactured and were scanned before the lamination process and after the lamination process to see if any differences could be noted. Although the spectra look similar, it can be seen that there is a slight intensity change in the peaks for foil 6 before and after lamination. This is a possible indicator that lamination processes has effected foil 6 and may have affected the bandgap within the structure leading to a more transparent product. Based upon the results from the transmission values seen in Table 3 this is very likely as foil 6 has proved to be the most transparent out of all the foils.

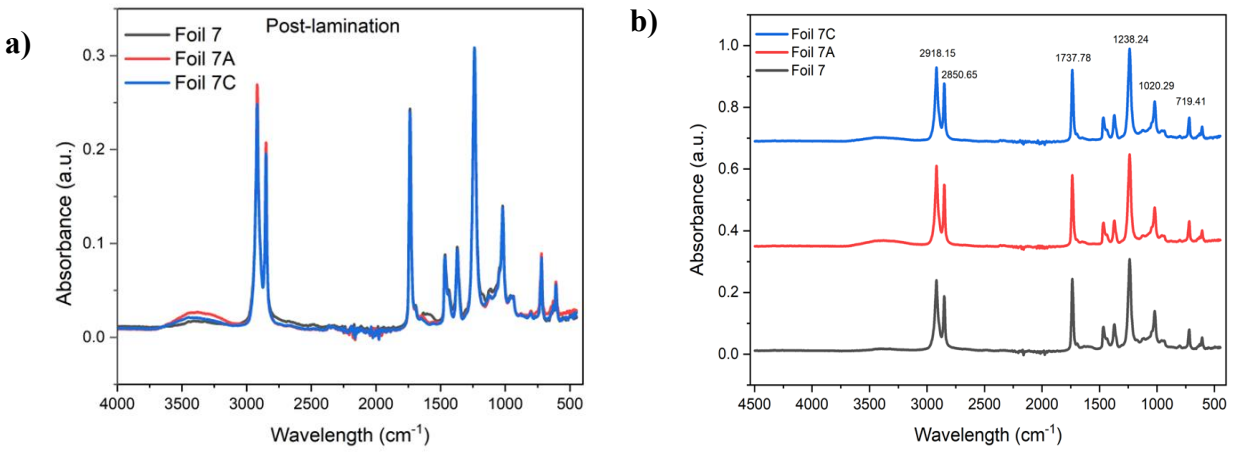


Figure 21. a) A graph comparing foil 7, 7A and 7C. b) A graph of foil 7,7A and 7C comparing the peaks.

The graphs in figure 21 look at comparing all of the foil 7's to see if they can be distinguished and to see if there are any differences based on the concentration added. It can be noted in graph a) that although the peaks are nearly identical there is an intensity in the peaks seen around 2900 cm^{-1} for foil 7A. This is likely due to the difference in concentration between foil 7 and 7C. Graph b) was used to help identify functional groups and it was found that all the peaks present were expected based upon the composition of foil 7. The similarity between the spectra for the three foils also highlights the fact that the foils were made with the same method and there was no contamination or change that would have hindered the results from the tests. The peak at 1737.78cm is indicative of a C=O bond from the EVA, and the peak at 2918.15cm is indicative of a C-H bond from EVA also. Two more apparent peaks can be seen at 1020.29cm and 719.41cm, these peaks are due to the additive.

4 Discussion

4.1 Summary of findings

Ten foils were made and tested for this research. The purpose of each foil was to improve a certain property of EVA as an interlayer for glass production. One of the foils stood out and showed impressive results for all of the tests and this was foil 7. It was found to have a high adhesion, a high crosslinking rate and a very good transmittance value competing with the reference foil and exceeding it in the majority of the tests performed. This led to the re-making of foil 7 and testing it further by creating three new foils 7A, 7B, 7C, with the hope of matching or exceeding the results obtained by foil 7. It was found that foil 7C performed even better than foil 7 with results higher than previously found. However, in order to understand the true potential of all of the foils it would be prudent to re-make the foils and re-test them a number of times to see if the tests will yield the same results for each occasion. Also due to the time constraint, strength testing was not part of the testing process and is an essential test that needs to be run. It is very important that the glass products that we buy are strong and can withstand large impacts. Therefore, it will be a very important test to conduct on the foils in the future to see if the foils would meet the standard requirements for everyday products.

4.2 Conclusions and future implications

Based upon the research conducted for this topic, it can be concluded that the experimentation and evaluation of the foils was an overall success. The research of each foil was aimed at improving certain properties such as the adhesion, the transmittance and the crosslinking percentage of the EVA foil already in commission and through fabrication and testing, a number of foils showcased significant improvements of the values, already deemed acceptable as the industry standard. The hope is that with the improvement of these properties the lifetime of the product can be increased and can become even more sustainable in harsher and more extreme climates such as places that experience heavy rain throughout the year or places that have harsh climates in the winter with a lot of snowfall and ice. Although much more testing and time must be invested into this research, it is a promising start into a field that is constantly being explored and new ideas are being developed and tested every day.

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