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Investigation of Epoxy Resin Spreading on Glass Surfaces

Haneen Nemer Al-Dahdooh

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Investigation of Epoxy Resin Spreading on Glass Surfaces

By

Haneen Nemer Al-Dahdooh

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THESIS EXAMINATION REPORT

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The Thesis Examination as A Partial Fulfillment of M. Sc. Degree in Materials Science and Engineering Was conducted on .......... Based on Examining the Thesis and the Students Presentation and the Subsequent Discussion, The Committee Recommends:

☐ Thesis is Satisfactory as is.
☑ Thesis is Satisfactory After Minor Modifications.
☐ Thesis should be Re-Evaluated After Major Modifications.
☐ Thesis is Rejected.

Examining Committee Members:

Thesis Supervisor: Name: Dr. Abdullatif Al-Matrouqi Signature: __________________________ Date: 8/3/2006

Member : Name: Dr. E. Al-Matrouqi Signature: __________________________ Date: 8/3/2006

Member : Name: Dr. Jan J. Scown Signature: __________________________ Date: 8/3/06

Approval of Program Coordinator:

Dr. Yousef Haik

Date: 11/3/06

APPROVAL:
Dean of Graduate Studies
Date: 18/4/06
Dedication

This thesis and all my future success are dedicated to very special people

To my beloved parents

Without their endless love, wisdom and guidance

None of this would have been possible
Abstract

The spreading of a small epoxy resin droplet (1.5μl) on a glass surface was examined with the objective of developing a new type of hydrophobic polymer coated glass which has the ability to reduce heat gain inside a building. The study covered three main aspects; the spreading kinetics of epoxy resin on a glass surface, the hydophobicity of the developed polymer-coated glass surface, and the reduction of solar radiation of this surface.

As wettability and spreading phenomena are a fundamental prerequisite for coating, the spreading kinetics of different curing agent concentrations (15:200, 18:200, 21:200) of epoxy resin on the glass surface (soda lime), were studied. All different curing agent concentrations of epoxy exhibit good spreading behavior. As the curing agent concentration of epoxy resin increases, the spreading kinetics on the surfaces decreases.

The hydrophobicity of the polymer-coated glass surface was tested and it was found that epoxy resin surface has the ability to repel water.

Dip-coating techniques provided a polymeric coated glass surface that had a transparent appearance giving the natural look of glass. This allowed the transmission of natural visible rays providing natural daylight. The developed polymer coated glass surface was found to reduce the heat absorbed due to solar radiation. A Kipp & Zonen Pyranometer CM11 in conjunction with a solar integrator were used for measuring the solar radiation absorbed by the glass surface. The results showed that the epoxy coated glass surface provided heat gain reduction ranges of between 23% and 60% depending on the solar irradiance.
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Chapter 1: Introduction and literature review
1.1 Introduction:

Wetting and dewetting processes play a very important role in a wide variety of industrial systems. They are a fundamental prerequisite for several applications, for example in paint films, composites, adhesives, printing, agricultural plant treatment, pharmaceutical tablets, cosmetics and coating.

Wetting comes from the word wet or wettable. When it is said that a substrate is wettable, the term "wettable" and its variations mean wettable by a liquid, i.e., the liquid spreads over the surface of a substrate.

Wettability or wetting is defined as the process in which a liquid spreads on a solid substrate. It can be estimated by determining the contact angle or the spreading coefficient. Figure 1.1 shows the wetting system, which consists of a liquid droplet, spreading on a solid substrate surrounded by a gas phase. The three phases (vapor, liquid and solid) reach equilibrium affected by their interfacial tensions. The relationship between the interfacial tensions $\gamma_{lv}$, $\gamma_{sv}$ and $\gamma_{sl}$ can be expressed by Young's equation (Young, 1805). This equation is listed below (Eqn. 1.1)

$$\gamma_{lv} \cos \theta = \gamma_{sv} - \gamma_{sl} \quad (1.1)$$

Where $\gamma_{lv}$, $\gamma_{sv}$ and $\gamma_{sl}$ refer to the interfacial energies of the liquid/vapor, solid/vapor and solid/liquid interfaces.
The wetting phenomenon could be defined relative to the macro and micro level as follows:

**Macroscopic definition of wetting:**

Full and complete coverage of the solid surface by the liquid

**Thermodynamic definition of wetting:**

Reduction in interfacial free energy when a liquid is placed in contact with a solid surface resulting in a contact angle $\theta < 90^\circ$. Complete spreading occurs when $\theta = 0^\circ$.

![Figure 1.1 Schematic of wetting system](image)
1.2 Wettability and spreading phenomena

1.2.1 Young's equation:

Young's equation was first discovered by the British scientist Thomas Young in 1805. Most wettability tests involve the measurement of contact angles of specific liquids on a surface. Young's equation stated in section 1.1 relates $\cos \theta$ and three interfacial tensions operating at the three phase line (solid, liquid and gas) (Young, 1805).

Based on Young's equation, $\theta$, can be defined by the mechanical equilibrium of a liquid drop under the action of interfacial tensions, as shown in Figure 1.2. Young's equation implies a single and a unique contact angle for a specific vapor-liquid-solid system.

According to Young's equation (Eqn. 1.1), the solid surface should have certain properties: (Kwok, 1998; Lam, 2001).

The surface should be

1. smooth
2. Homogenous
3. Rigid
4. Non deformable,
5. Inert with respect to the liquid used.

![Figure 1.2 Schematic of the mechanical definition of Young's equation. Horizontal balance of forces. Young's equation $\gamma_s \cos \theta = \gamma_{sv} - \gamma_{sl}$](image-url)
1.2.2 Contact angle:

Contact angle, $\theta$, is a quantitative measure of the wetting of a solid by a liquid. It is defined geometrically as the angle formed by a liquid at the three-phase boundary where a liquid, gas and solid intersect. When a liquid droplet interacts with a solid surface, the droplet attains an equilibrium shape. The droplet can be characterized by the contact angle. Figure 1.3 shows a schematic presentation of a liquid droplet on a solid substrate.

![Figure 1.3 Schematic of contact angle](image)

If the angle $\theta$ is less than 90°, the liquid is said to wet the solid. If it is greater than 90°, it is said to be non-wetting. A zero contact angle represents complete wetting. Figure 1.4 shows the schematic of a drop on a solid surface with different values of contact angles.
Contact angle

\[ \cos \theta \]

\[ \begin{array}{c|c|c|c}
\theta & 0^\circ & 90^\circ & 180^\circ \\
\hline
\cos \theta & 1 & 0 & -1 \\
\end{array} \]

Figure 1.4 Schematic of drop on solid surface showing dynamics of spreading from complete wetting, partial wetting to incomplete spreading

The contact angle of the droplet has a direct relationship with the interfacial tensions of the three phases. This relation is presented by Young's equation Eqn.1.1, stated in Section 1.1 (Young, 1805).

Contact angle has an important role in many other fields, such as coatings (Adamson, 1997), composites and polymers (Adamson, 1997), adhesives (Ashleya et al., 2003), biomedical applications (Kasemo, 2002; Ashleya et al., 2003), biological Processes (Neinhuis and Barthlott, 1997), oil recovery (Adamson, 1997; Drummond and Israelachvili, 2002), and many different engineering and environmental applications (Sasa, 2003). Another important and specific use of contact angle is to calculate the interfacial tensions of solid phase interfaces of the wetting system (Kwok, 1998).
1.2.2.1 Static & dynamic contact angle:

A static contact angle is produced when the three-phase boundary is not in motion. The value of the static contact angle depends on the recent history of the interaction. When the drop has expanded on the substrate, the angle is called the "advanced" contact angle. When the drop has contracted, the angle is called the "receded" contact angle. These angles fall within a range, with advanced angles approaching a maximum value and receded angles approaching a minimum value.

A dynamic contact angle is produced when the three phase (liquid/solid/vapor) boundaries is in motion and these angles are referred to as "advancing" and "receding" angles.

The difference between 'advanced' and 'advancing', 'receded' and 'receding' is that, in the case of static angles, motion is incipient, and in the case of dynamic angles, motion is actual. Dynamic contact angles may be tested at various rates of speed. Dynamic contact angles measured at low velocities should be equal to the measured static angles.

1.2.2.2 Utilization of contact angle data:

The most important use of contact angle is in describing the wetting phenomenon of solid/liquid interactions. Contact angle is used as the most direct measure of wetting. Other experimental parameters may be derived directly from contact angle and surface tension results. Some examples are:
Work of adhesion: it is the work needed to separate the liquid and solid phases, or it is the negative free energy associated with the adhesion of the solid and liquid phases. It is used to express the strength of the interaction between the two phases. It is given by the Young-Dupre equation as:

$$W_a = \gamma (1 + \cos \theta)$$  \hspace{1cm} (1.2)

Work of cohesion: it is defined as the work required to separate a liquid into two parts; it is a measure of the strength of molecular interactions within the liquid (Girifalco and Good, 1957) and it is given by:

$$W_c = 2 \gamma$$  \hspace{1cm} (1.3)

Work of spreading: the negative free energy associated with a liquid spreading over a solid surface (Girifalco and Good, 1957). It is given as:

$$W_s = \gamma (\cos \theta - 1)$$  \hspace{1cm} (1.4)

Wetting tension: a measurement of force/length (Girifalco and Good, 1957). It is defined as:

$$\tau = \frac{F_w}{P} = \gamma_{LV} \cos \theta$$  \hspace{1cm} (1.5)

This value, wetting force normalized for length, also represents the product of the cosine of the contact angle and the surface tension. It indicates the strength of the wetting interaction without separate measurement of surface tension.
1.2.2.3 How is contact angle measured?

There are two different approaches for measuring the contact angles of non-porous solids, goniometry and tensiometry. Goniometry involves the observation of a sessile drop of test liquid on a solid substrate. Tensiometry involves measuring the forces of interaction as a solid comes into contact with a test liquid.

1.2.3 Contact angle hysteresis

Basing on Young's equation stated in section 1.1, one would expect to find only one value of wetting contact angle for a real solid/liquid drop/gas system. However, in a real system, even in static conditions, at least two different contact angles can be measured on the same liquid and on the same solid surface, which are termed as advancing and receding contact angles.

The difference between the maximum (advanced/advancing) and minimum (receded/receding) contact angle values is called contact angle hysteresis. This phenomenon was discovered 80 years ago (Shulman, 1919).

Contact angle hysteresis can be calculated using equation 1.6 listed below:

\[ H = \theta_a - \theta_r \]  

(1.6)

Where \( \theta_a \) is the advancing contact angle and \( \theta_r \) is the receding contact angle.
Many studies and research have gone into analysing of the significance of hysteresis. It has been used to characterize surface heterogeneity (Marmur, 1994; Decker and Garoff, 1996), surface roughness (Oliver and Mason, 1980) and mobility or deformation of a solid surface (Neumann, 1974; Li, 1991).

1.2.4 Spreading coefficient

Another quantitative measure of wetting is the spreading coefficient, which is defined as the energy difference between the solid substrate and the contacting gas and liquid phases. The relationship of the spreading coefficient can be written in terms of surface free energies, and is defined for a liquid-solid–vapor system (Myers, 1999) as:

\[ S_{1s} = \gamma_{sv} - (\gamma_{lv} + \gamma_{sl}) \]  \hspace{1cm} (1.7)

Where \( S_{1s} \) refers to the spreading coefficient for the system, and \( \gamma_{sv}, \gamma_{lv}, \gamma_{sl} \) refer to the interfacial energies for the system. For complete spreading where \( \theta = 0 \), the spreading coefficient \( S = 0 \); and for partial spreading where \( 0 < \theta < 90 \), the spreading coefficient \( S < 0 \).
1.2.5 Dynamic of wetting:

When a liquid is placed in contact with a solid surface; two possible wetting regimes can occur:

1.2.5.1 Complete wetting:

In complete wetting systems (Figure 1.7.a); the liquid spreads completely over the solid surface to form a flat film resulting in a zero contact angle ($\theta = 0$) and spreading coefficient $S = 0$. Alteraifi and Sasa (2003) studied the spreading kinetics of liquid droplets on solid substrates. They applied silicon oil as a liquid polymer on a glass surface. The silicon oil exhibited complete spreading independent of the liquid's viscosity. Figure 1.5 shows the spreading kinetics of silicon oil on a glass surface (soda lime) (Alteraifi and Sasa, 2003).

1.2.5.2 Incomplete wetting

In Figure 1.7.b, the liquid spreads over the solid surface, the contact angle $0 < \theta < 90$, and the spreading coefficient $S < 0$. Many studies of partial wetting have been carried out by Brochard-Wyart and de Gennes (1992), Van Remoortere and Joos (1993) and de Conick et al. (2001). The spreading of glycerol liquid on a glass surface represents an incomplete wetting system. Figure 1.5 shows the spreading kinetics of glycerol on glass presented as contact area verses spreading time for 1.5µL drop volume.
Non wetting occurs if the liquid rests on the solid surface with a high contact angle that is greater than 90°, (θ > 90°). Figure 1.7.c presents the schematics of non-wetting.
The symptoms of incomplete wetting as related to coating are:

1. Non-uniform shapes, resulting in voids

2. Less than optimum adhesion

3. Reduction in mechanical properties and reduction in durability

Figure 1.6 was created by Intelligent System Laboratory Group in Michigan University (1999); it shows two levels of wetting problems: macro and micro problems. The image on the left is magnified 20X; the right hand image is magnified 100X. The images show a single polyurethane sized fiber tow. This sizing is not compatible with the vinyl ester resin being used. At low magnification, voids resulting from air trapped between bundles can be seen. These voids are macroscopic; they are much larger than the fiber's diameter. Also visible in these images is a high degree of light reflection due to scattering. This occurs because there is no molecular level wetting between resin and fiber. At the higher magnification, this effect is seen as bright areas (Intelligent System Laboratory Group, USA, 1999).
Figure 1.6 Macro & molecular level of wetting problem (Intelligent System Laboratory Group, USA, 1999)
Dynamics of wetting
\[ S = \gamma_{sv} - (\gamma_{lv} + \gamma_{sl}) \]

a) \( S \) (spreading coefficient) = 0
\( \theta \) (contact angle) = 0
**Complete wetting**

b) \( S \) (spreading coefficient) < 0
\( \theta \) (contact angle) < 90
**Partial wetting**

c) \( S \) (spreading coefficient) > 0
\( \theta \) (contact angle) > 90
**Non wetting**

Figure 1.7 Schematic of a) complete wetting = 0, b) Partial wetting \( 0 < \theta < 90^\circ \), c) non wetting \( \theta \geq 90^\circ \)
1.3 Characterization of the solid surface

The contact angle measurements give data related to the thermodynamics of a liquid/solid interaction. However, surface tension measurements give data reflecting the thermodynamics of the liquid tested.

To characterize the wetting behavior of a particular liquid/solid interaction, the contact angle must be reported. But to characterize the thermodynamics of the solid surface itself more analysis is required. Various methods are used but the same basic principle applies for each. The solid is tested against a series of liquids and contact angles are measured. Calculations based on these measurements produce a parameter (critical surface tension or surface free energy), which quantifies the characteristics of the solid and mediates the properties of the solid substrate.

1.3.1 Surface free energy and critical surface tension

Surface free energy is defined as the work required to increase the area of a substance by one unit area. The surface free energy of a solid is sometimes also referred to as the "surface tension" of the solid substrate. For example; water has a very high value of surface tension because it has a high degree of hydrogen bonding. Organic molecules with polar groups, such as iodide and hydroxyl, have a slightly lower surface energy than water. Pure hydrocarbons are even lower.
The critical surface tension, or the surface free energy, is a property of the solid in the same way as the surface tension is of a liquid. Many different approaches are used to determine the solid surface energy. These approaches are listed below:

1. An approach that depends on contact angle measurements (Zisman, 1964; Fowkes, 1964; Oss et al., 1988).

2. Direct force measurements (Derjaguin et al., 1980; Pashley et al., 1988; Fogden and White, 1990).

3. Solidification front interaction with particles (Chen et al., 1977).

4. Film flotation (Fuerstenau et al., 1990).

5. Sedimentation of particles (Van-Butler et al., 1987).


7. Gradient theory (Guermeur et al., 1985).

8. The Lifshitz theory of Vanderwaals forces (Israelachvili, 1972; van Giessen et al., 1997).

Among these approaches, the most preferable one is contact angle measurement.
1.3.2 Zisman plots and critical surface energy

Measuring surface free energy is very difficult and many approaches have been developed to determine it. One approach to enable the determination of surface free energy was developed by Zisman at Naval Research Laboratory (Zisman, 1964). The plot was made by plotting the cosine of the contact angle versus the surface free energy of various wetting liquids on a given solid. The resulting plot is a straight line. Thus, there exists some unique value for each polymeric solid where the cosine of the contact angle is unity. This value is termed the critical surface free energy. A liquid with surface free energy below the critical value will wet and spread over the solid surface, whereas a liquid with surface energy above the critical value might wet but won't spread. Figure 1.8 presents Zisman plots of the contact angles of various homologous series on solid Teflon.
Using the concept of critical surface energy, it is possible to characterize a wide variety of polymers and correlate the critical surface energy with polymer structure. Fluorinated materials have low values of critical surface energy. Hydrogenated materials, such as polyethylene and polypropylene, have slightly higher values. For other substituents (Cl, O, N), the critical surface free energy is a little higher (Shaw, 1992). From the critical surface energy data just given, fluorinated and hydrogenated materials will be among the least wettable.

Figure 1.8 Zisman plots of the contact angles of various homologous series on solid Teflon at 20°C. •, n-alkanes; ○, other hydrocarbons; ■, esters and ethers; □, halocarbons and halohydrocarbon; △, different liquid
1.3.3 Effect of temperature on surface tension

There are many factors that affect the spreading of liquid on a solid surface and affect the surface tension. One of these factors is temperature; the surface energy is inversely proportional with temperature. This means that surface energy decreases with increasing temperature (de Ruijter et al., 1998). Figure 1.9 shows the surface energy of an organic liquid as a function of temperature (de Ruijter et al., 1998). The surface tension reaches to zero at boiling point.

![Figure 1.9 The effect of temperature on the surface tension (Intelligent System Laboratory Group, USA, 1999)](image-url)
Figure 1.10 represents the effect of temperature on some polymeric liquids. The surface tension decreases as time increases. However, as polymers don't boil, the surface tension never reaches to zero. The surface energy decreases with temperature, but not as it does for small molecule liquids. Polymer structure also affects surface energy. Lower surface energy polymers have weakly interacting atoms in the backbone, whereas high values arise from strong interactions between atoms in polar materials.

Figure 1.10 Surface tension vs. temperature for polymer melts and liquids: o, polychloroprene; ●, polyisobutylene; ▲, poly(n-butyl methacrylate); □, poly(vinyl acetate); ■, polydimethylsiloxane (Intelligent System Laboratory Group, USA, 1999)
1.3.4 Theory of Adhesives:

Petrie (2000) introduced important theories of adhesion to represent the bonding mechanism between molecules in materials. The adsorption theory of adhesion states that "Adhesion results from molecular contact between two materials and the resulting surface forces that develop" (Petrie, 2000). The process of establishing intimate contact between an adhesive (liquid) and the adhered (solid substrate) is known as wetting. Adhesion results from the forces of molecular attraction such as van der Walls forces" (Petrie, 2000).

In his experiment; Petrie investigated the spreading of epoxy \( \gamma = 47 \text{ dynes/cm} \) over different surfaces with different values of surface tension. He provides experimental results showing that Polymers with low energy surfaces, such as polyethylene and fluorocarbons, are not good adhesives and they are difficult to bond with adhesives.

Figure 1.11 shows good and poor wetting of epoxy spreading over different surfaces. Good wetting occurs when the liquid (adhesives, coatings, etc ....) spreads out over the substrate in a uniform film and make a low degree contact angle between the substrate and the adhesives (Figure 1.11. epoxy adhesive on metal substrate).
Poor wetting occurs when the adhesive forms droplets on the surface making a high degree contact angle (Figure 1.11 epoxy adhesive on fluoroethylene propylene substrate).

Based on Petrie's results, we can come up with the following:

For any liquid (adhesive, coating, ...) to wet a surface, the liquid should have a lower surface tension, $\gamma$, than the solid's surface energy (or critical surface tension), $\gamma_c$.

For good wetting and resulting strong adhesion forces: $\gamma_{\text{adhesive}} \ll \gamma_c$ substrate

For poor wetting and low adhesion forces: $\gamma_{\text{adhesive}} \gg \gamma_c$ substrate

Figure 1.11 Spreading of epoxy on different surfaces (Petrie, 2000)
1.3.5 Hydrophilic and hydrophobic surfaces as they relate to contact angle

- **Hydrophilic surfaces**

A hydrophilic surface is a water absorbent surface. The liquid transfers from the syringe onto the surfaces, almost completely emptying the syringe of the liquid. Liquid spreads rapidly across the surface in a thin layer. The droplet is characterized by having a low contact angle; $\theta < 90^\circ$. The drops can be irregular and can spread into neighboring drops, as shown below in Figure 1.12.

![Low contact angle](image)

*Figure 1.12 Liquid drops spreading on hydrophilic surface (Corning Company, New York, USA)*

- **Hydrophobic surfaces**

With hydrophobic surfaces, less liquid transfers onto the surface. Liquid volume deposited onto the slide is variable. Liquid attaches to the substrate in a smaller area. The droplet is characterized by a high contact angle $\theta > 90^\circ$, very round spot morphology, inconsistent liquid volume deposited and spots may move from their original placement, as shown below in Figure 1.13.
Optimal Surface

Up to a point, increasing contact angle changes transferred liquid volume very little and spots become increasingly uniform, small and round. Under optimal conditions, consistent liquid volumes transfer to the slide surface and form uniformly round spots. The droplet has excellent surface area for probe attachment; excellent morphology and the drop remains in place, as shown in Figure 1.14.
1.4 Coatings on glass

The deposition of a coating on a solid generates new interfaces between dissimilar materials and involves a serious consideration of wettability and spreading. It was found that wettability promotes adhesion through physical, chemical and mechanical bonds acting across coating-substrate adhesion.

A previous study related wettability, spreading and interfacial phenomena with coating at high temperatures (Asthana and Sobazak, 2000).

Large area coatings on glass have been used in a wide variety of products, such as plate glass for architectural and automotive glazing, hot and cold end coatings for container glass and for other types of glass, like lamps. The most common material that can be used in wet coatings is commercially available organic polymer materials. Wet coating can generate highly functional coating materials with excellent properties for glass surfaces.

The materials used in wet coating can be either transparent or non-transparent materials. Moreover, in wet coating there is the chance to make materials with unique, interesting and excellent properties and while utilizing cost-effective coating techniques. Some interesting material properties are easy-to-clean properties, antifogging properties, strength increasing properties on thin glass, micro-patternability of coatings, photochromic or electrochromic properties. Other coatings include antireflective (AR) coatings, IR reflecting coatings, coloured coatings or conductive coatings.
1.4.1 Coating technologies

As general prerequisites for obtaining wet chemical coatings with high optical qualities on glass, it can be stated that the coating process step has to be carried out under clean room conditions, the coating liquid has to be filtered and the glass has to be cleaned properly. The most well-known wet coating techniques are listed below:

- Dip coating techniques

Dip coating can be described as a process where the substrate to be coated is immersed in a liquid and then withdrawn at a well-defined withdrawal speed under controlled temperature and atmospheric conditions (Scriven, 1988). The coating thickness is mainly defined by the withdrawal speed, by the solid content and the viscosity of the liquid. If the withdrawal speed is chosen such that the shear rates keep the system in the Newtonian regime, the coating thickness can be calculated by the Landau-Levich equation (1942). (Eqn. 1.8).

\[
    h = 0.94 \cdot \frac{\eta \cdot \nu^{2/3}}{\gamma_{LV}^{1/6} \cdot (\rho \cdot g)^{1/2}}
\]

Where:

- \( h \) = coating thickness,
- \( \eta \) = viscosity,
- \( \gamma_{LV} \) = liquid-vapour surface tension
- \( \rho \) = density,
- \( g \) = gravity

(Eqn. 1.8)
As shown by James and Strawbridge (1986) for an acid catalyzed silicate sol, thicknesses obtained experimentally match calculated thickness very well. One advantage part of dip coating processes is that by choosing an appropriate viscosity the coating thickness can be varied with high precision from 20 nm up to 50 μm while maintaining high optical quality. The schematics of a dip coating process are shown in Figure 1.15.

![Figure 1.15 Stages of the dip coating process: a) dipping of the substrate into the coating solution, b) wet layer formation by withdrawing the substrate and gelation of the layer](image)

Another type of dip coating is realized when the atmosphere controls the evaporation of the solvent and the subsequent destabilization of the sols by solvent evaporation, leading to a gelation process and the formation of a transparent film due to the small particle size in the sols (nm range) (Brinker et al., 1990; Brinker, Hurd, and Ward, 1988). This is schematically shown in Figure 1.16.
Dip coating processes are used for plate glass for solar energy control systems and anti-reflective coatings on windows (Schröder, 1969). The dip coating technique is also utilized for optical coatings, e.g. on bulbs, for optical filters or dielectric mirrors by various SMEs and other companies, fabricating multilayer systems with up to 30 or 40 coatings with very high precision. More recently, an angle-dependent dip coating process has been developed (Arfsten et al., 1997, Dislich, 1986). Figure 1.17 shows the angle-dependent dip coating process.
- **Spray coating techniques**

Spray coating techniques are widely used in industry for organic coating. For coating irregularly shaped glass forms like pressed glass parts, lamps or container glass (cold end coating) it is also feasible (Arfsten et al., 1997). Spray coating, produces fine droplets leading to very homogeneous coatings on hard substrates, but the coating material does not hit the surface in the form of liquid droplets but more or less in the form of dried small particles in the nanometer range. Due to the high reactivity of these particles when reaching the hot surface, a continuous glass film can be formed.

- **Flow coating processes**

Flow coating can be described as the process where the liquid coating is poured over the substrate to be coated, as shown schematically in Figure 1.18. The most common application of flow coating techniques is the outfitting of automotive glazing from polycarbonate with hard coatings, but they can also be used for float glass to apply functional coatings (Belleville and Floch, 1992). The coating thickness depends on the coating liquid viscosity, the angle of inclination of the substrate and the solvent evaporation rate. The advantage of the flow-coating process is that non-planar large substrates can be coated easily.
**Spin coating process**

In the spin coating process, the substrate spins around an axis which should be perpendicular to the coating area. The spin-on process has been developed for production of so-called spin-on glasses in microelectronics and substrates with a rotational symmetry, e.g. optical lenses or eye glass lenses (Scriven, 1988). The schematics are shown in Figure 1.19.

---

**Figure 1.18 Scheme of the flow coating process (Schmidt and Mennig, 2000)**

**Figure 1.19 stages of the spin process: deposition of the sol, spin up, spin off and gelation by solvent evaporation (Schmidt and Mennig, 2000)**
Fully automated spin coating processes have been introduced in the ophthalmic glass industry under clean room conditions and fully automated handling. The coating thicknesses vary between several hundreds of nanometers and up to 10 micrometers.

The thickness of a spin coated layer depends on the angular velocity, velocity and solvent evaporation rate. This is expressed by a semi-empirical equation (Meyerhofer, 1978).

\[
  h = \left( 1 - \frac{\rho_A}{\rho_{A0}} \right)^{1/3} \left[ \frac{3}{2} \frac{\eta \cdot m}{\rho_{A0} \cdot \omega^2} \right]^{1/3}
\]

\( \rho_A \) = mass of volatile solvent per unit volume , \( \rho_{A0} \) = initial value of \( \rho_A \) , \( h \) = final thickness , \( \eta \) = viscosity , \( \omega \) = angular speed , \( m \) = evaporation rate of the solvent.

Since \( m \) has to be determined empirically any way, the more simple formula given in Eqn 1.10 may be used:

\[
  h = A \cdot \omega^B
\]

Where \( A \) and \( B \) are constants to be determined empirically. Lai, Chen and Weill (Lai, 1979; Chen, 1983; Weill, 1986) independently determined the thickness of films obtained by spin coating, using different angular speeds, and their results fit very well with equation 1.10. \( B \) was determined to be in the interval between 0.4 and 0.7, which is in rather good agreement with Eqn. 1.9, where the exponent for \( \omega \) is 0.67.
• **Capillary coating**

Spray and spin coating processes are characterized by the fact that the coating material cannot all be brought onto the substrate. In spray coating processes, more than 100% overspray is used, and similar amounts are wasted with spin coating.

Dip and flow coating processes are dependent on the shelf life of the coating material and in optical dip coating only 10 to 20 percent of the coating liquid can actually be used for the fabrication of coatings. To overcome these problems, the so-called capillary or laminar flow coating process was developed by Floch (Chen1983; Weill1986) and CONVAC Co., which combines the high optical quality of the dip coating process with the advantage that all the coating liquid can be exploited. In Figure 1.20 the capillary coating process is shown schematically.

![Figure 1.20 Schematics of the capillary coating process (Floch, 1990)](image-url)
Coating thicknesses of as little as 15 μm in high optical qualities are obtained, as well as multilayer coatings for dielectric mirrors.

- **Roll coating**

Roll coating processes on plate glass are not state-of-the-art techniques. However, pilot plant investigations have shown that optical qualities are obtainable (Belleville, and Floch, 1992). It is important to note that in order to avoid structures on the surface, cutted roles have to be used, and coating thickness and viscosity of the liquid have to be adapted very thoroughly. Using cutted roles, the amount of liquid transported onto the glass surface is defined by the voids cut out of the role. After the deposition, the parts have to coagulate and to form a homogeneous film. For this reason, the wetting behaviour of the glass against the liquid has to be perfect and the drying speed has to be adapted to the film forming velocity. Therefore, temperature and atmosphere have to be controlled perfectly.

- **Printing technique**

The most common printing technique for glass decors is the silk screen printing process (Floch, and Priotton, 1990).
This state-of-the-art technology is widely used (automotive industry, decorative glasses e.g. in dashboards and windows of kitchen stoves). Coating materials based on organic polymers, to be cured at low temperatures or by UV curing, as well as enamel coatings with ceramic paints and appropriate low melting glass frits are used. Enamel-like coatings are densified near the $T_g$ of the glass substrate or, in combination with a thermal strengthening process or with a bending process, at appropriate higher temperatures. Typical film thickness is in the range from several 10 to several 100 $\mu$m. Therefore the coefficients of thermal expansion of enamel have to be matched to that of the substrate glass. Printing coating technology is presented in Figure 1.21.
- **Chemical coating**

Chemical coatings can be described as a process where a chemical reaction occurs, e.g. the reduction of a metal. The most common process is the fabrication of mirrors where the glass surface acts as a nucleating agent for the reduction of $\text{Ag}^+$ to $\text{Ag}^0$ in presence of the reducing agent. The vast majority of all mirrors are still fabricated using this process.

- **Drying and curing techniques**

Drying and curing techniques are important for obtaining the appropriate coating properties. Depending on the type of coating material, high temperature curing and low temperature curing can be distinguished. If high chemical durability is required, a coating temperature just below $T_g$ of the glass is chosen in order to maintain the shape. Glass frits or sol-gel systems are then converted to ceramic- or glass-like coatings. If special functions have to be obtained, the control of the atmosphere may be of importance.
1.4.2 Types of coating material related to hydrophobicity and hydrophility

- **Hydrophobic coatings**

A hydrophobic coating is a coating that has the ability to repel water. Most hydrophobic coatings are based on organic polymers and can be employed by dip or very special spray coating techniques (Terrier et al., 1995).

These types of hydrophobic coating have many advantages:

- They are easy to clean because they repel water.
- They show good mechanical properties and stability.
- These coatings show a permanent hydrophobicity and dust repelling on car windshields.

From Central Glass Company a process has been introduced by Terrier and Roger (1995) where sol-gel derived ZrO₂ is deposited on plate glass by dip coating and treated at low temperatures to form micro- or nanoporous layers. These layers are then impregnated by perfluorinated silanes. Embedded in the zirconia network, the perfluorinated side chains show a high thermostability and "survive" the bending process. Another type of hydrophobic coating has been developed by PPG (Agrawal, et al., 1988).
• *Hydrophillic coatings*

Hydrophillic coatings can be defined as coatings with a good wetting behavior in the presence of water. This type of coating is needed in many automotive applications. And are used both outside and inside the vehicles.

Hydrophillic coating materials based on organic polymers exhibit several disadvantages.

- Poor mechanical properties (scratch and abrasion resistance).
- Poor stability against wet climate conditions & UV radiation.
- Tendency to swell by incorporation of water.

In order to overcome these disadvantages, functionalized inorganic-organic nanocomposites known for their good adhesion and excellent scratch resistance with hydrophilic compounds, have recently been developed (Yahiaoui et al., 1999).
1.4.3 Testing and examining hydrophobic coated surfaces

*Micro-Droplet Hydrophobicity* measures to what degree a surface repels water. The contact angle is measured by the inside angle that is formed between the surface and a line is drawn at a tangent to the side of the water droplet.

![Contact Angle - Low](image1)

Contact Angle - Low
Solid Surface Energy - High
Wetability and Adhesion - Good

![Contact Angle - 90°](image2)

Contact Angle - 90°
Solid Surface Energy - Moderate
Wetability and Adhesion - Fair

![Contact Angle - High](image3)

Contact Angle - High
Solid Surface Energy - Low
Wetability and Adhesion - Poor

*Figure 1.22 Degree of hydrophobicity*

Channel Master Company in USA and Germany has developed a hydrophobic reflector coating to overcome the problems that are related to harsh environmental conditions, such as high temperature, high humidity, wind and other factors such as snow, ice and rain which accumulate on the reflector's surface causing problems in transmission and reception of signals.

The Channel Master hydrophobic reflector coating consists of a solid layer of fluorocarbon polymer, and polyurethane is applied to an antenna surface as shown in Figure 1.23. This chemical process yields a tough, durable film with superior ultraviolet resistance and the ability to repel water and ice over a long term. In addition, the hydrophobic reflector coating process provides resistance to corrosion, abrasion, chemical attacks and impacts.
Moreover, Channel Master Company tried not only to create a hydrophobic coating but also a super hydrophobic coating, which means that when a water droplet comes into contact with the reflector surface, it repels it. The droplet forms a high contact angle on the hydrophobic treated surface that creates a shape more closely resembling a sphere. This spherical shape allows minimal contact with the surface, so the water droplet rolls off like a ball. Figure 1.24 shows water droplets on a) an untreated surface and b) on a hydrophobic surface.
A second study on the hydrophobic coating was carried out by United States Patent in 1998; they invented a multilayered hydrophobic window glass to improve resistance to climatic stresses such as humidity, UV radiation and shrinkage due to heat change; and also to mechanical stresses such as, friction due to windshield wipers and cleaning rags. According to their invention, a window glass consisting of a substrate made of glass is covered by one or more hydrophobic-oleophobic, abrasion-resistant coatings comprising a mineral sublayer and a hydrophobic-oleophobic layer, wherein the density of the sublayer is equal to at least 80% of that of its constituent material.
Braunsberger and Dziubek carried out research on the damaging effects of water drop corona on insulating materials. Earlier investigations focused on silicon rubber which is intrinsically hydrophobic. Now the research work was extended to epoxy resin systems. Water drops are put on epoxy substrate and they are exposed to an increasing AC field. (Braunsberger, Dziubek, Kurrat, 2003) Figure 1.25 a shows a water drop on a hydrophobic cycloaliphatic epoxy (HCEP) and Figure 1.25 b shows a water drop on standard cycloaliphatic epoxy (CEP) for outdoor applications. Figure 1.26 shows the new hydrophobic epoxy insulator (Braunsberger, Dziubek, Kurrat, 2003).

![Figure 1.25 Water drop of 30 ml on surface of a) HCEP b) CEP (Braunsberger, Dziubek, Kurrat, 2003)](image)

![Figure 1.26 Construction of modern epoxy insulator (Braunsberger, Dziubek, Kurrat, 2003)](image)
1.5 Heat transfer through glazing

Heat is usually transferred from the higher temperature object to the lower temperature object (Holman, 2002; Bejan, 1993). The thermal performance of windows is of great concerns due to energy loss through glazing, which can amount a considerable expense in heating and air-conditioning. Heat transfer through glazing is mainly due to radiation, conduction and convection (Holman, 2002).

Radiation

Radiation is heat transfer by the emission of electromagnetic waves which carry energy away from the emitting object (Holman, 2002). For ordinary temperatures (less than red hot), the radiation is in the infrared region of the electromagnetic spectrum. The relationship governing radiation from hot objects is called the Stefan-Boltzmann law (Holman, 2002; Bejan, 1993).

\[
P = e \sigma A (T^4 - T_c^4)
\]  

(1.11)

\[
P = \text{net radiated power} \quad e = \text{emissivity (=1 for ideal radiator)}
\]

\[
A = \text{radiating area} \quad T = \text{temperature of radiator}
\]

\[
\sigma = \text{Stefan's constant} \quad T_c = \text{temperature of surroundings}
\]

\[
\sigma = 5.6703 \times 10^{-8} \text{ watt/m}^2\text{K}^4
\]
Radiant heat energy is transmitted through single-, double- and triple-glazing, causing heat loss in winter and heat gain in summer. Radiation occurs when heat, after being sent through space, travels to a distant object where it can be reflected, absorbed or transmitted. One way to minimize this effect is to use a reflective film (i.e., heat mirror) or a reflective coating (i.e., low emissivity, or Low E) on the glazing. Low E glass and invisible films, both transparently coated to maintain visibility, will reflect a large percentage of radiant heat energy that would otherwise escape. Similarly, outward radiant heat energy from the sun, which causes excessive summer heat gain, is reflected outward (Holman, 2002).

Figure 1.27 The Sun at 5800K and a hot campfire at perhaps 800 K give off radiation at a rate proportional to the 4th power of the temperature (Nave, 1999)
Conduction

Conduction occurs when energy passes from one object to another (Holman, 2002). Conduction is both a cause of heat loss through sealed insulating glass units and the primary cause of heat loss through window sashes and frames. The thermal insulation value (R value) of sealed insulating glass units can be improved by increasing the number of glass panels and air spaces; for example, from double to triple, or by filling the units with gas.

For heat transfer between two plane surfaces, such as heat loss through the wall of a house, the rate of conduction heat transfer is (Holman, 2002; Bejan, 1993):

\[
\frac{Q}{t} = \frac{KA(T_{\text{hot}} - T_{\text{cold}})}{d} \tag{1.12}
\]

- \(Q\): heat transfer in time \(t\)
- \(K\): thermal conductivity
- \(A\): area.
- \(d\): thickness of barrier
- \(T\): temperature

Figure 1.28 The conduction heat mechanism
Convection

Convection results from the upward movement of warm, light air currents. When gases (including air) are heated, they expand, become less dense, and rise. As the gases cool, they increase in density and fall. These convection currents carry heat energy from warmer to cooler areas (i.e., from the warm interior to the cold exterior of window glazing) (Holman, 2002; Bejan, 1993).

Figure 1.29 Heat transfers by convection mechanism (Nave, 1999)
1.5.1 Heat gain control for windows

In the past, before innovations in glass, films, and coatings, a typical residential window with one or two layers of glazing allowed roughly 75-85% of the solar energy to enter a building (Klems, 1989), which has a negative impact on summertime comfort and cooling bills, especially in hot climates.

The weak thermal characteristics of windows became a prime target for research and development in the attempt to control the indoor temperatures of buildings. This led to the development of many types of glass that control heat gain and loss, reduce glare, minimize fabric fading, provide privacy, and occasionally provide added security in windy, seismic, and other high-hazard zones (Klems, 1989). New construction and window replacement applications commonly use glazing with these coatings. Some of these types of glass are listed below (Lyons and Reardon, 2005):

- **Tinted or "toned" glass** is the oldest of all the modern window technologies and most common type of absorbent glass. Under favorable conditions, it can reduce solar heat gain by 25% to 55%. Tinted glass is made by alteration of the chemical properties of the glass. Both glass and plastic laminate may be tinted. The tints absorb a portion of the sunlight and solar heat before it can pass all the way through the window into the building.
- **Reflective glass** has either a vacuum-deposited metal coating or a pyrolytic coating. Vacuum-deposited coatings are soft and must be glazed facing indoors. Pyrolytic coatings are hard and durable and can be glazed facing outdoors. Where glare may annoy neighbours, reflectivity should be kept below 15 to 20 percent.

- **Spectrally selective glazing** is commonly used for cooling climates or for westerly elevations where solar control and natural lighting are a priority. Spectrally selective glazing maximises light transmission while simultaneously reflecting unwanted solar radiation (UV and near infrared). Spectrally selective coatings can also have low emissivity. Spectrally selective tints reduce infrared light (heat) transmission while allowing relatively more visible light to pass through (compared to bronze- or gray-tinted glass). For buildings that use daylight for lighting, a spectrally selective window is a good choice. Spectrally selective glass also absorbs much of the ultraviolet (UV) portion of the solar radiation.

- **Polymers** are used instead of glass in some applications, such as translucent glazing and skylights. Plastic glazings may also be included in composite laminates to improve impact resistance or within double glazing to improve insulation.
• **Single glazing** offers little resistance to the passage of heat. The small amount of insulation that single glazing does provide is actually due to thin films of still air that exist next to the glass (Klems and Keller, 1987).

• **Double-glazing** offers much better insulation. It comprises two panes of glass with a sealed space between. The space is filled with air or an inert gas with better insulating properties than glass (Klems and Keller, 1987).

The best thermal performance for air-filled units occurs when the space between the panes is about 12 mm (Klems and Keller, 1987). If the double glazing is also to be used for noise reduction, a wider gap may be appropriate with some trade-off in thermal performance.

![Diagram of a double glazed window](Image)

*Figure 1.30 Double glazed window (Lyons and Reardon, 2005)*
For best performance, solar control glass should be used for the outer pane and low emissivity glass for the inner pane. The solar control glass prevents unwanted solar radiation entering, while the low emissivity glass reduces heat loss from inside. The low e-glass also blocks heat radiated from the outer pane of glass when it heats up.

A low cost alternative to conventional double-glazing is to use a thin, flexible, transparent polyethylene membrane in place of the inner pane of glass. The membrane is attached to the window frame using a high quality, transparent tape and is shrunk taut using a hairdryer.

This system can provide performance similar to that of plain, clear double-glazing at a much lower cost. It can be applied to almost any glazed fixture that has a frame.

Low emissivity (low-e) glass has a coating that allows short wavelength energy (daylight) from the sun to pass into the house but reduces the amount of the long wavelength energy (infrared heat) that can escape through the window. That is why this type of glass is often called a "Oheat mirror". Some low-e coatings and solar control films reduce solar heat gain without impairing visible light transmission excessively. These include tinted glass and spectrally selective coatings, which transmit visible light while reflecting the long-wave infrared portion of sunlight.
Low-e and reflective coatings usually consist of a layer of metal a few molecules thick. The thickness and reflectivity of the metal layer (low-e coating) and the location of the glass it is attached to directly affect the amount of solar heat gain in the room. Most window manufacturers now use one or more layers of low-e coatings in their product lines. Any low-e coating is roughly equivalent to adding an additional pane of glass to a window. Low-e coatings reduce long-wave radiation heat transfer by 5 to 10 times. The lower the emissivity value (a measure of the amount of heat transmission through the glazing), the better the material reduces the heat transfer from the inside to the outside. Most low-e coatings also slightly reduce the amount of visible light transmitted through the glazing relative to clear glass. Here are representative emissivity values for different types of glass (Lyons and Reardon, 2005):

- Clear glass, uncoated: 0.84
- Glass with single hard coat low-e: 0.15
- Glass with single soft coat low-e: 0.10

A pyrolytic coating baked on at a high temperature constitutes a "hard coat" low-e coating. These are often made of a metallic oxide. One layer is about 1/10,000 the diameter of a human hair. "Soft coat" low-e coatings are applied to the glass at lower temperatures and even thinner thicknesses than hard coatings. Both types of low-e coatings (within insulated glazing assemblies) are typically warranted for 10 to 50 years.
The only spectrally selective coatings now available are modified soft coat low-e coatings. The selective properties of the coatings are determined by modifying the coating's thickness and number of layers. A spectrally selective tinted glazing with a pyrolytic hard coat serves a similar purpose (Lyons and Reardon, 2005).

**Solar Comfort Control Company** in USA (2001) has designed and developed a low cost solution to prevent infrared solar heat and ultraviolet light rays from entering all types of glass and glass-like surfaces. The film is applied onto the substrate using an advanced glass coating spray technique that replaces high cost processes like vacuum deposition. The new coating provides tremendous savings in both heating and cooling for commercial and residential buildings. The heat gain through the gold coating is about 45%-60%.

**Solar Comfort Control Company** succeeded in the production of uniform, tightly adhered, thin, transparent gold coatings, and, in particular, in the efficient and economical production of such films on surfaces. More particularly, this invention relates to the production of thin transparent blue to blue-green gold coatings on nonmetallic surfaces using an electroless spraying process for deposition resulting in films which transmit a high degree of visible light, while still blocking a large amount of infrared light. The physical properties of transparent gold films have include the transmittance of significant amounts of visible light while blocking much of the infrared light from passing through the substrate.
The gold coating on a glass window works as follows; during the summer, visible light is allowed through the substrate, while heat producing infrared and dangerous UV light ray is “stopped” from entering the window, and the air-conditioned cool air is kept inside, decreasing costs. During the winter, the heat inside the room is reflected back, decreasing heating costs (Solar Comfort Company, USA, 2001).

Moreover, glass maybe coated by a polymeric material; for example, polyvinyl butyrate (PVB) or an adhesive PVB is conventionally used in safety glass laminates. It is used to reduce heat gain and loss inside the building. Polymeric films generally last about 8 to 10 years before they start looking worn.
1.5 Objectives of thesis:

In this thesis, new hydrophobic polymer-coated glass is developed using dipping coating techniques. Good wettability and spreading of epoxy resin on glass surfaces promotes good coating on a glass substrate. This thesis has three objectives:

1. To investigate the spreading of epoxy resin on glass by performing critical experiments to examine the spreading mechanism of epoxy resin on a glass surface.
2. To test the hydrophobicity of new epoxy coated glass created by dipping techniques. This could be useful in the cleaning process of glass.
3. To measure the solar radiation absorbed by coated glass, based on its solar radiation ratio, and to compare these results with the results obtained with the uncoated glass.
Chapter 2:

Materials and Methods
2.1 Liquid polymer

- Epoxy resin:

The basic material which is used in this study as a coating material applied on glass is a modified Diglycidyl of Bisphenol A (DGEBA) epoxy resin. The resin is supplied by Dow Chemicals as DER324 and used as received. Epoxy resin is defined as a molecule containing more than one epoxide group. The epoxide group is also termed oxirane or ethoxyline group. Epoxy resin has been used in many applications, since it was first synthesized in the mid 20th Century. It is used in construction, aerospace and more recently in medical applications (Meier, 1995). The most common applications of the resin are high solids coating, flooring compounds, grouts, adhesives, decoupage coatings and composites (Garter et al., 2003). The commercial resin in itself is a formulated blend of DER 3331 (DGEBA) and a reactive diluent C12-C12 aliphatic glycidyl and it is used in three different blend ratios (curing agent concentrations) 21:200, 18:200 and 15:200. Figure 2.1 shows the structure of DGEBA epoxy and C12-C14 Aliphatic Glycidyl Ether.
A) DGEBA Resin

\[
\begin{array}{c}
\text{H}_2\text{C}=	ext{CH}-
\end{array}
\]

\[
\begin{array}{c}
\text{O} \\
\text{R}---\text{O}---\text{CH}_2-\text{CH}---\text{CH}_2
\end{array}
\]

\[R = \text{C}_2\text{H}_2\text{O} \text{ to } \text{C}_1\text{4} \]

B) C\text{12} to C\text{14} Aliphatic Glycidyl Ether

The ratio of mixing was calculated based on the following equation:

\[
\text{Phr of hardener} = \frac{\text{Hardener eq.wt} \times 100}{\text{Epoxide .wt of resin}}
\]  

(2.1)

Phr: parts - by weight - per 100 parts resin.

EQ.wt: weight - in grams - which contains one gram equivalent of material.
Table 2.1 shows the epoxide equivalent weights for the hardener and the epoxide which are 21 g and 200 (average) respectively. The epoxide equivalent weight is defined as the weight in grams which contains one gram equivalent of epoxide (Encyclopedia of Polymer Science and Engineering, 1985).

<table>
<thead>
<tr>
<th>Material</th>
<th>Wt. (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hardener DEH20</td>
<td>21</td>
</tr>
<tr>
<td>Epoxy DER 324</td>
<td>200</td>
</tr>
</tbody>
</table>

Table 2.1 Epoxide equivalent weights for hardener and epoxide

Epoxy resin belongs to a family of thermosetting polymers, meaning that once it is cured, the coating will not tend to soften at higher temperatures. It achieves its beneficial properties as a result of a heat catalyzed chemical reaction. Then it reacts to form complex cross-linked polymers.

Epoxy resin was selected in this study because it has excellent properties (Murphy, 1998):

- It has considered to be a good coating material (most paints and coatings are thermoset polymers to avoid reshaping when exposed to natural and artificial weathering)
- Cured at room temperature 25°±3°
- Excellent resistance
- High mechanical properties
- Low shrinkage percentages
- Non reactive with solid surface
- A coating must provide a continuous film that will: resist penetration by salt ions; resist the action of osmosis, adhere to and expand/contract with the substrate; resist breakdown from weathering and exposure; and be flexible and durable enough for handling.

The physical and chemical properties of epoxy resin are presented in Table 2.2 (Dow Chemical Company).

<table>
<thead>
<tr>
<th>Properties</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Appearance</td>
<td>Liquid</td>
</tr>
<tr>
<td>Color</td>
<td>Pale yellow</td>
</tr>
<tr>
<td>Boiling point</td>
<td>Decomposes prior to boiling</td>
</tr>
<tr>
<td>Freezing point</td>
<td>May solidify $&lt;-15^\circ\text{C}$</td>
</tr>
<tr>
<td>Water solubility</td>
<td>$&lt;1%\text{wt (25 }^\circ\text{C)}$</td>
</tr>
</tbody>
</table>

Table 2.2 Physical & chemical properties of Epoxy resin
2.2 Solids

- *Soda-lime glass*

Soda lime glass is a common type of window glass. It has been used as a substrate in the experiment to test the spreading of epoxy resin droplet on it. In this study soda lime slides with an area of (75x25 x 1 mm^3) were obtained from Menzel-Glaser Company Geschitten, Germany.

The most common type of soda lime glass is a float glass. In this type of glass the glass is cooled in a molten tin, enriching its float side with tin oxide.

Soda lime glass contains about 13% sodium oxide, which is highly soluble in water and reacts to form sodium hydroxide, and this gives the glass its hydrophilic property. The formation of sodium hydroxide causes a reaction with the carbon dioxide in air, leading to the formation of white sodium carbonate powder on the glass surface. Glass surfaces can catch germs, dusts and chemicals very easily, therefore a strict method of cleaning the glass and keeping it in a vacuumed condition is needed (Berg, 1993).

There is a wide range of cleaning procedures that render the glass wettable and free of particles (Lel, 1997; Pul, 1984). An important point in all glass cleaning procedures is that the glass surface must be used or coated as soon as possible after cleaning (Pul, 1984). Glass surfaces have a high surface energy. They have a tendency to absorb particulate germs and organic contamination from the ambient environment; and this creates unacceptable defect. An adsorbing of organic contaminant will generate a heterogeneous wettability and this may lead to non uniform coating.
Glass cleaning steps:

- Immerse the glass slides in a high concentration of chromic acid solution (K2Cr2O7+H2SO4) for 4-6 hours.
- Wash with distilled water.
- Rinse with acetone.
- Insert the slides in a vacuum oven for 1/2 an hour at 90°C.
- Leave the glass slides in a vacuum to cool.
- Transfer the slides into a dissector, where they are stored.
- Clean glass could be used in the experiment.

The glass substrates were scanned using Scanning Electron Microscopy techniques (SEM) before being cleaned and after. Figures 2.3 and 2.4 show the difference between the uncleaned and cleaned glass substrate.
Figure 2.2 SEM of uncleaned glass (before cleaning process) showing grime and impurities.

Figure 2.3 SEM image of clean glass showing no contamination or particles on the surface
2.3 Experimental procedures

2.3.1 Preparation of the epoxy resin:

The epoxy resin and curing agent were stirred homogenously for 10 minutes in a vessel. The total amount of resin prepared was 30 grams to assure uniformity.

The vessel was left for 5 minutes to remove the air bubbles to the surface. Then the liquid was charged in the syringe to test the spreading of the liquid.

This experiment was repeated for three different curing agent ratios: 21:200, 18:200, 15:200.

2.3.2 Dynamic contact area measurement procedure

Figure 2.4 shows the experimental setup. To start the experiment, epoxy was charged into a 5 μl syringe obtained from Hamilton Company, Switzerland. The syringe was attached to a metal stand and mounted vertically by means of a micromanipulator on the top of the substrate. The micromanipulator is used to carefully adjust the position of the needle tip of the syringe above the clean slide. The tip of the syringe was positioned a few micrometers from the surface of the glass to eliminate the effect of impact when the droplet was released. The droplet volume was selected to be 1.5 ml so the gravity effect is negligible. Using Bond number as:

\[ Bo = \frac{\rho g R_o^2}{\sigma} \]  

(2.2)
Where $g$ is the gravity acceleration, $R_o$ is the radius of the spherical droplet before spreading and $\rho$ is the liquid density. The bond number is 0.32 for the liquid used in this study. Since $Bo << 1$, capillary forces were expected to dominate gravity.

After a single use of the syringe, it is cleaned by immersion in acetone to dissolve the liquid inside it. Then it is transferred to the oven to allow the acetone to dry. Before conducting the experiment, the syringe is flushed with epoxy resin once or twice. At least nine measurements were taken for epoxy on glass slides.

A CCD digital video camera (JVC TK-c1380, Japan) with 10x eyepiece magnification was positioned underneath the glass substrate. The camera was connected to a video recorder, which was in turn connected to an image analysis system (Analysis Soft Imaging System, GmbH ltd, Germany). At the top, there was a light source, which was ring light fiber optics (Model LG-R66.OLYMPICS-JAPAN). It was used to avoid the heat produced by an ordinary bulb light. Heat may affect the surface tension of the liquid and produce a blurred image. The experiment was carried out under ambient conditions, i.e., Temperature $=25 \pm 1 ^\circ C$ and $47\% \pm 3\%$ RH.
Figure 2.4 The experimental setup for contact area measurement
The contact area was measured as a function of time. Figure 2.5 shows the image frame that was taken by the imaging analysis software. The area to be measured was indicated in the overlay by a circle.

Figure 2.5 The contact area in mm$^2$ for cured epoxy over glass substrate measured by software after 10 seconds.

Software and calibration (dynamic contact area)

Image processing:

A calibration was done using calibration sample image analysis software (Analysis 3.0 Soft Imaging System GmbH Germany March 1999).
Calibrate input

1. A suitable calibration specimen is placed above the digital camera with 10x magnification.
2. Acquire the focused and enhanced image from the microscope.
3. Snapshot the image.
4. From the image menu select the Configure input.
5. Select XY calibration tab and insert the required magnification.
6. From Set unit, define the calibration distance unit.
7. Select the vertical option for the calibration distance to be defined.
8. Insert the calibration length according to the selected distance as defined by the calibration sample, then save the calibration by clicking on Save.
9. Add new calibration to the magnification table by clicking Add then press Ok.

2.3.3 Glass coating

The glass slides were dipped in the mixture, which covered all the slides, resulting in a total coated surface of 1800mm². Then the glass slides were withdrawn from the vessel to be inserted in an oven at 80 °C, resulting in a transparent glass. The coated glass has a thin film of epoxy resin coated onto the inner and outer surfaces of the glass (25 mm x 75 mm). The coated glass has the ability to reduce the temperature inside the building (minimize the heat gain).
2.3.3 Measuring heat gain by solar radiation for uncoated and epoxy-coated glass.

In this experiment, a Kipp & Zonen Pyranometer CM11 and a solar integrator were used to measure the solar radiation absorbed by the glass surface. The Kip & Zonen pyranometer CM 11 is designed to measure the irradiance (radiant flux, Watt/m²) on a plane surface, which results from direct solar radiation and from the diffuse radiation incident from the hemisphere above.

Because the CM 11 exhibits no tilt dependence, it can measure solar radiation on inclined surfaces as well. Reflected solar radiation can be measured in the inverted position. As shown in figure 2.6 the pyranometer is composed of a hemisphere. The most common application for this device is testing materials and high accuracy solar monitoring.

![Figure 2.6 Kip & Zonen pyranometer CM 11.](image)
Applications of pyranometer CM 11:

- High accuracy solar monitoring

- Road weather station

- Material testing

The Kipp & Zonen Solar Integrator shown in Figure 2.7 was used in conjunction with pyranometer CM11 to measure the solar irradiance in W/m². Figure 2.8 shows the experimental setup of heat measurement.
2.8 Experimental setup of heat measurements

The first measurement taken was of solar radiation $E_{\text{sun}}$ in W/m$^2$, and then the glass slides were positioned one by one above the hemisphere of the pyranometer. The readings were taken for un-coated glass as $(E_{\text{un-coated}})$, followed by coated glass with different curing agent concentrations of 21:200, 18:200 and 15:200 $(E_{21}, E_{18}, E_{15})$. The first solar radiation ratio was calculated as energy absorbed by the coated glass surface over the energy absorbed by uncoated glass.
Chapter 3:

Results and Discussion
3.1 Spreading kinetics of cured epoxy related to contact ratio:

The spreading of epoxy resin on a glass surface was examined with respect to the measurement of the contact area. The contact area was then converted to the contact ratio (A). Figure 3.1 shows a schematic of a liquid drop on a solid surface, where \( R_c \) is the radius of the drop-solid substrate contact area, \( \rho \) is the radius of the curvature and \( h \) is the height of the drop with respect to the surface.

The contact ratio \( A^* \) is defined as the ratio between the contact area of the spreading liquid drop on a solid to the surface area of the liquid sphere before spreading (Alteraifi and Herbawi, 2005).

\[
\text{Contact ratio (A*)} = \frac{A_c}{A_s} \quad (3.1)
\]

Where \( A_c \) is the contact area of the liquid drop on the solid surface.

\[
A_c = \pi R_c^2 \quad (3.2)
\]

![Figure 3.1 Schematic of drop on solid substrate](image)
$A_s$ represents the surface area of the drop before spreading.

$$A_s = 4\pi R_o^2$$  \hspace{1cm} (3.3)$$

Where $R_o$ is the radius of the drop’s sphere before spreading.

The relationship between contact angle and contact ratio can be derived from the definition of the contact ratio based on the spherical cap approximation relation as given in (Eqn.3.4), as follows:

$$\theta = \frac{4V}{\pi R_c^3}$$  \hspace{1cm} (3.4)$$

Where $V$ is the drop volume given as:

$$V = \frac{4\pi R_o^3}{3}$$  \hspace{1cm} (3.5)$$

One should mention that the spherical cap approximation (Eqn.3.4) can only be applied to low contact angle values, where $\theta < 90^\circ$. Substituting Equations 3.2 and 3.5 in equation 3.4 and applying the definition of contact ratio Eqn.3.1, $A^*$ reduces to be a function of $\theta$ only;

$$A^* = \left(\frac{3}{2}\theta\right)^{-2/3}$$  \hspace{1cm} (3.6)$$

or

$$\theta = \frac{2}{3} (A^*)^{-3/2}$$  \hspace{1cm} (3.7)$$
3.1.1 Spreading kinetics of epoxy with curing agent concentration of 21:200 on glass surface

Figure 3.2 shows the spreading kinetic of cured epoxy with a curing agent concentration of 21:200 on glass surfaces. The figure presents the contact ratio ($A^*$) versus time (min). The experimental results were considered for 35 minutes. After that the epoxy was cured (crosslinked) at which time the contact ratio reached a constant value of 1.93. Initially, the contact ratio increases with time. The spreading of epoxy 21:200 on the glass surface shows three different regions. The first was the spontaneous region that presented as region (A), where a high rate of spreading occurred. The second region was the slow spreading region (B), which may have been due to competing forces of the cross linking of the epoxy resin and the spreading forces. The transition from region A to B was indicated at the inflection point. The third region (C) is where the epoxy resin cross-linked and the spreading ceased.

The contact angle $\theta$ in degrees based on Eqn 3.6 is plotted against time in seconds for epoxy with a curing agent concentration of 21:200 in Figure 3.3. The duration of the experiment was 35 minutes. The figure shows that the contact angle decreases with time. The initial contact angle was $45.70^\circ$. After 35 minutes, the contact angle reached a constant value of $14.3^\circ$. 
The rate of change of contact ratio (dA*/dt) is plotted against time, as shown in Figure 3.4. The rate of change of A* decreased rapidly in the first 100 seconds of spreading. The trend of the data suggests that the transport of epoxy resin ought to enter the slow spreading region with the start of polymer crosslinking. This point, where the liquid moves from the spontaneous region A to the slow spreading region B, is indicated as the inflection point. For epoxy resin 21:200 this point is found after approximately 90 seconds (approximately 1.5 minutes).
Figure 3.3 Kinetics of spreading of cured epoxy 21:200 on glass surface

Figure 3.4 Rate of spreading of cured epoxy 21:200 on glass surface
3.1.2 Spreading of epoxy with curing agent concentration of 18:200 on glass surface

A similar analysis was carried out to examine the spreading kinetics of epoxy with a curing agent concentration of 18:200 on glass surfaces (soda lime surfaces). The spreading kinetics is presented as spreading time against contact ratio, as shown in Figure 3.5. The duration of the experiment was 50 minutes. After that, the epoxy was cured (cross linked). It was found that the contact ratio increases with time. After a period of 50 minutes, the contact ratio becomes independent of time and reaches a constant value of 2.08. Similar to the spreading of epoxy 21:200 on a glass surface, the spreading of epoxy 18:200 on the glass surface showed three different regions. The first was the spontaneous region, presented as region (A), where a high rate of spreading occurred. The second region was the slow spreading region (B), which may have been due to competing forces of the cross linking of the epoxy resin and the spreading forces. The point, where the liquid moves from the spontaneous region A to the slow spreading region B, is indicated as the inflection point. The third region (C) is where the epoxy resin cross-linked and spreading ceased. Figure 3.6 shows the contact angle $\theta$ in degrees versus time in seconds for cured epoxy with a curing agent concentration of 18:200. The figure shows that the contact angle decreases with time. It was found that the initial contact angle was 46°. After 50 minutes, the contact angle reached a constant value of 12.8°.
Figure 3.7 shows the rate of change of contact ratio (dA*/dt) versus time. The rate of change of A* decreased rapidly in the first 100 seconds of spreading. From Figure 3.7, the inflection point defined in section 3.1.2, is found after approximately 120 seconds (approximately 2 minutes).
Results & Discussion

Figure 3.6 Kinetics of spreading of cured epoxy 18:200 on glass surface

Figure 3.7 Rate of spreading of cured epoxy 18:200 on glass surface
3.1.3 Spreading kinetics of epoxy with curing agent concentration of 15:200 on glass surface

A similar analysis was carried out for the spreading of cured epoxy with a curing agent concentration of 15:200 on glass surfaces (soda lime surfaces) and the results are presented in Figures 3.8, 3.9 and 3.10. The contact ratio is plotted against time in minutes in Figure 3.8. The experimental results were considered for 90 minutes, after which the epoxy was cured (cross linked). It was found that the contact ratio reaches a constant value of 2.3.

The contact angle $\theta$ in degrees based on Eqn 3.6 is plotted against time in seconds for cured epoxy with a curing agent concentration of 15:200 in Figure 3.9. The duration of the experiment was 90 minutes. The figure shows that the contact angle decreases with time. The initial contact angle was $46.2^\circ$. After 90 minutes, the contact angle reached a constant value of $10.9^\circ$. 
The rate of change of contact ratio (\(dA^*/dt\)) is plotted against time as shown in Figure 3.10. The rate of change of \(A^*\) decreased rapidly in the first 100 seconds of spreading. The inflection point is found after approximately 180 seconds (approximately 3 minutes).

Figure 3.8 Kinetics of spreading of cured epoxy 15:200 on glass surface; region A is the spontaneous region, B is the slow spreading region and C is the epoxy crosslinking region.
Results & Discussion

Figure 3.9 kinetics of spreading of cured epoxy 15:200 on glass surface

Figure 3.10 Rate of spreading of cured epoxy 15:200 on glass
Figure 3.11 summarizes the spreading kinetics of epoxy resin with different curing agent concentrations (uncured epoxy, 15:200, 18:200, 21:200) on a glass surface. The spreading kinetics is presented as the contact ratio ($A^*$) value of epoxy resin plotted against time. The experimental measurements were interrupted after 35 minutes for epoxy (21:200), after 50 minutes for epoxy (18:200), after 90 minutes for epoxy (15:200) and after 130 minutes for uncured epoxy. The experimental conditions were similar, i.e. the same solid surface type used (glass) and the same liquid surface tension ($\gamma_{lv} =$47 dyne/cm for epoxy resin) applied in the same conditions for the vapor-liquid system, while varying the curing agent concentration. Thus, Figure 3.11 demonstrates that the spreading kinetics is independent of the epoxy curing agent concentration. The contact ratio ($A^*$) increases exponentially as the contact angle decreases with time. A similar analysis was reported by Lau and Burns (1973). They investigated the spreading kinetics of polymer melts on plane solid surfaces, namely; polystyrenes of narrow molecular weight distribution on glass. The spreading process was observed over temperatures ranging from 110-260°C and for nominal average molecular weights of polystyrene varying from 2000 to 37,000 (Lau and Burns, 1973). The liquid-solid contact area increased as the contact angle decreased with time, both in an exponential fashion.
The previous results are summarized in Table 3.1. The table presents the measured contact area, contact ratio and contact angle for different concentrations of the curing agent.

The inflection points vs. epoxy curing agent concentration is presented in Figure 3.12. The figure shows that the inflection point increases as the curing agent decreases, i.e. it takes a longer time for epoxy to crosslink with lower concentrations of the curing agent.
Results & Discussion

Figure 3.12 The effect of curing agent concentration on the inflection point

Table 3.1 The spreading parameters of cured epoxy on glass surfaces.
The relationship between solidification time (the time needed for the epoxy to completely cross-link) and the curing agent concentration in the resin is presented in Figure 3.13. Epoxy with a curing agent concentration of 21:200 solidifies after 35 ± 3 minutes from the time of releasing the droplet from the syringe. A longer time is needed for epoxy with a curing agent concentration of 18:200, which solidifies after 50 ± 5 minutes from the release of the droplet from the syringe. 15:200 epoxy solidifies after 90 ± 10 minutes. Figure 3.13 shows that as the curing agent concentration decreases, the epoxy spreading takes longer time to solidify.

![Curing agent concentration of epoxy](image)

**Figure 3.13 Solidification time of cured epoxy.**

After examining the wettability and spreading kinetics of epoxy resin on glass surface, it can be concluded that epoxy resin as a polymeric material exhibits a good spreading behavior reporting a low contact angle which is consistent with the results observed by Petrie (2000) for epoxy spreading on different surfaces.
3.2 Epoxy coating on glass at different temperatures

As general prerequisites for obtaining wet coatings with high optical qualities on glass under clean room conditions, the coating liquid has to be filtered and the glass has to be cleaned properly.

In this study, the glass slides were completely dipped in the mixture, resulting in a total coated surface area of 1800 mm². Then the glass slides were withdrawn from the vessel and inserted in an oven at a temperature of 80°C to obtain a transparent coating on the glass (amorphous polymer instead of opaque crystalline polymer).

Figure 3.14 shows images taken by a digital camera magnified 10 times of epoxy with different curing agent concentration on glass at a room temperature of 25°C and at 80°C. The temperature affects the curing process (cross linking) of epoxy resin, leaving a specific pattern on the surface. At room temperature, the cured epoxy left a translucent nearly to opaque coating as shown in Figures 3.14 D, E and F. At 80°C, the obtained coating was transparent with a smooth surface that allows the transmission of visible rays and enhances natural daylighting, as presented in Figures 3.14 A, B and C. Thus at temperature of 80°C, the epoxy coated glass surface is better than that at room temperature.
A) Epoxy 21:200 coating on glass surface at temperature $= 80^\circ$C

B) Epoxy 18:200 coating on glass surface at temperature $= 80^\circ$C

C) Epoxy 15:200 coating on glass surface at temperature $= 80^\circ$C

D) Epoxy 21:200 coating on glass surface at room temperature $= 25^\circ$C.

E) Epoxy 18:200 coating on glass surface at room temperature $= 25^\circ$C

F) Epoxy 15:200 coating on glass surface at room temperature $= 25^\circ$C

Figure 3.14 Images of epoxy coated glass magnified (10 x) at different temperatures.
3.3 The hydrophobicity of epoxy resin

The presence of liquid film or dust alters the visibility of window glass and reduces its transparency. To overcome such difficulties, the coated glass surface should exhibit a hydrophobic property so that it becomes non-wettable.

A hydrophobic surface is defined as a surface that has the ability to repel water. This experiment was conducted to examine if the epoxy resin can be considered as a hydrophobic or hydrophilic surface. The spreading of water drops on coated and uncoated glass surfaces was examined to test the ability of these surfaces to repel water.

In Figure 3.15, we show the spreading of a water droplet on epoxy coated glass. In this experiment, the syringe was positioned at a distance from the glass slide, as shown in Figures 3.15a, b. Then the syringe was brought into contact with the glass surfaces (Fig 3.15c). Figure 3.15d shows the water droplet 5 sec after the syringe touches the glass surface. Finally, Figure 3.16e shows the water droplet 20 sec after releasing the droplet from the syringe.
Results & Discussion

Figure 3.15 The spreading of water droplets on epoxy-coated glass.

Figure 3.16 a) shows a water droplet spreading over an uncoated glass surfaces. A water droplet resting on the epoxy-coated glass surface and forming a high contact angle is shown in Figure 3.16 b).

Figure 3.17 shows the maximum contact area of a water droplet on a) uncoated glass surface, b) 21:200 epoxy-coated glass surface, c) 18:200 epoxy-coated glass surface and d) 15:200 epoxy-coated glass surface. The experimental measurements were interrupted after 50 seconds.
Figure 3.16 Images of water droplet; a) water droplet on glass surface (left), and b) water droplet on epoxy coated glass (right)

Figure 3.17 Images of water droplet; maximum contact area for water droplet on a) uncoated glass surface, b) 21:200 epoxy-coated glass surface, c) 18:200 epoxy-coated glass surface and d) 15:200 epoxy coated glass surface
The spreading behavior of water droplets on epoxy-coated glass, with different curing agent concentrations, and un-coated glass is presented in Figure 3.18. The figure shows the spreading kinetics as contact ratio ($A^*$) against time of a water drop on uncoated glass surface, epoxy 21:200 coated glass, epoxy 18:200 coated glass and epoxy 15:200 coated glass.

Figure 3.18 shows the spreading kinetics of a water drop on an uncoated glass surface (soda lime surface). The experiment was interrupted after 30 seconds. The contact ratio increases with time, reaching a constant value of 1.27 (contact area = 7.97 mm$^2$).
This corresponds to a contact angle of a value of 26.84°. Similar results were reported by Alteraifi and Sasa (2003), who investigated the spreading of water on a soda lime surface.

Water exhibits a low contact angle on glass due to the very high value of surface tension of water as a result of its high degree of hydrogen bonding. In addition, a glass surface is considered to be a hydrophilic surface which has a greater ability to absorb water than other surfaces. The spreading kinetics for a water drop on an epoxy-coated glass surface with a curing agent concentration of 21:200, 18:200 and 15:200 are presented in Figure 3.18. All experiments were interrupted after 30 seconds. The epoxy repels the water and forms a high contact angle and small contact ratio A*. Contact ratios were found to be 0.57, 0.52 and 0.48, which correspond to contact angle values of 88.8°, 102.2°, and 139.46° respectively.

The contact ratio of water spreading on an uncoated glass surface was found to be at least twice as high as the contact ratio of water spreading on epoxy coated glass. The decrease in the contact ratio is about 50% less for the epoxy-coated surface.
However, our experimental setup limitations can not detect the differences in the spreading area of the water droplet on glass surface for the three epoxy curing agent concentrations (Fig 3.19). The experimental results show that epoxy resin is a hydrophobic polymer coating. This helps in the self cleaning process and allows good visibility through the glass surface, which agrees with the results observed by Braunsberger (2003) for epoxy coating as a hydrophobic coating.
3.4 The measurement of heat gain by solar radiation for coated and uncoated glass:

Windows and other glazed external surfaces have a major impact on the energy efficiency of the building envelope. If not designed correctly they allow unwanted heat transfer between the indoors and the outdoors. If designed correctly, they will help in maintaining high comfort levels inside the buildings (Holman, 2002).

Heat is usually transferred from the higher temperature object to the lower temperature object. There are three modes of heat transfer: radiation, conduction and convection (Holman, 2002; Bejan, 1993). The major and the most effective heat gain through glazing results from solar radiation. In this study, the direct and diffused (indirect) solar radiation absorbed by the coated and un-coated glass, were measured. The output readings for the coated glass and uncoated glass present an indicator for thermal changes that occur in the case of epoxy coating glass.

In this experiment, a Kipp & Zonen Pyranometer CM11 was used in conjunction with solar integrator to measure the solar radiation absorbed (radiant flux in Watt/m²) by the glass surface. The first measurement taken by pyranometer CM11 gives the direct and indirect solar radiation in W/m². This value is defined as \( E_{\text{sun}} \). The solar radiation absorbed by uncoated glass is presented as \( E_{\text{uncoated}} \). Then, the coated glass slides were positioned one by one above the hemisphere of the pyranometer. The amount of solar radiation absorbed by coated glass with different curing agent concentrations (21:200, 18:200 and 15:200) are defined as \( E_{21}, E_{18}, E_{15} \), respectively.
The solar radiation ratio (SRR) is defined as the solar radiation absorbed by the coated glass over the solar radiation absorbed by uncoated glass. This ratio provides a value to compare different types of glass and to recognize the changes due to the applied coating. This ratio is given by:

\[
\text{Solar radiation ratio (SRR)} = \frac{\text{Solar radiation absorbed by coated}}{\text{Solar radiation absorbed by uncoated glass}} \tag{3.8}
\]

The solar radiation ratio (SRR) is a dimensionless quantity and normalized in order to allow comparison between the obtained results.

The solar radiation absorbed by coated and uncoated glass in watts per square meter versus solar radiation absorbed by the sun \(E_{\text{sun}}\) is presented in Figure 3.23.

The solar radiation was measured by pyranometer CM11 in different environmental conditions (cloudy and sunny days) and at different times of the day for several days.

The averages for these values were measured under the following conditions:

\[-E_{\text{sun}} = 781 \text{ w/m}^2 \text{ at 11:00 am}\]
\[-E_{\text{sun}} =1010 \text{ w/m}^2 \text{ at 1:00 pm}\]
\[-E_{\text{sun}} =94 \text{ w/m}^2 \text{ at 3:00 pm}\]
\[-E_{\text{sun}} =77 \text{ w/m}^2 \text{ at 5:00 pm}.\]
For each of the above mentioned values, the solar radiation absorbed by the coated and uncoated glass was measured, indicating four different values for solar radiation (SR).

The maximum solar radiation (SR) had an average value of 1010 w/m² which was measured from 12:00 to 2:00 pm. The lowest solar radiation (SR) had an average value of 77 w/m², which was measured from 4:00-5:00 pm. Figure 3.23 shows that the solar radiation absorbed by uncoated glass surface has absorbed a higher amount of solar radiation than the epoxy-coated glass surface. The solar radiation absorbed by coated glass was found to decrease as the curing agent concentration decreases.

Figure 3.20 Solar radiations effect on coated and uncoated glass surface at different values of $E_{sun}$
Figure 3.21 Solar radiation ratio for coated and uncoated glass surface

Figure 3.24 presents the solar radiation ratio vs. different types of coated and uncoated glass. The solar radiation ratio SRR for uncoated glass is normalized, which indicates that 100% of solar radiation is absorbed by the uncoated glass.

The solar radiation absorbed by epoxy 21:200 coated glass is about 77%, which indicates a heat gain reduction of 23% as compared with uncoated glass. The solar radiation absorbed by epoxy 18:200 coated glass is about 66%, which reduces heat gain by 34%. The solar radiation absorbed by epoxy 15:200 coated glass is about 40% which reduces heat gain by 60%. It can be concluded that the glass with the epoxy coating can reduce heat gain resulting from solar radiation through glazing. The experimental results show that the solar radiation absorbed by the epoxy-coated glass surface is reduced in the range of 23%-60%.
Chapter 4

Conclusion and summary
The spreading of epoxy resin, with different curing agent concentrations of 21:200, 18:200, 15:200 on a glass surface, was examined. The contact ratio $A^*$ was used as a measurement of wettability supported with the result of the contact angle. Epoxy resin exhibits a good wettability over a glass substrate, reporting low contact angles. Epoxy coating was applied to glass, using dip-coating techniques at room temperature, then at a temperature of 80°C, to obtain a transparent uniform coating. Water droplet spreading was examined over the epoxy coated glass, revealing a hydrophobic surface with a high contact angle, which is a property that helps in the self-cleaning process of glass and allows the rain to slip over the glass substrate.

The solar radiation absorbed by epoxy-coated glass and uncoated glass was measured using pyranometer CM11. The results obtained were fruitful. The epoxy-coated glass was able to reduce heat gain due to solar radiation by 23%-60% compared with the uncoated (clear) glass. The heat gain, through single uncoated glass is 100% and heat gain due to solar radiation through epoxy-coated glass ranges from 40% - 77%. The three types of epoxy-coated glass exhibit a good wettability, hydrophobicity and the ability to reduce the heat gain due to solar radiation inside the building. However; the epoxy with a curing agent concentration of 15:200 exhibits the best results with the lowest contact angle, which equals 10.88°, and a contact ratio with a value of 2.31. It has a transparent appearance, the highest degree of hydrophobicity with a water contact angle of 139.5° on a glass surface, and 60% heat reduction when compared with the uncoated glass.
Chapter 5

Future Work
Glass is a very important material in the facades of buildings. It has a major impact on the energy efficiency of the building envelope. Therefore, the performance, safety and durability of windows should be taken into consideration. The study of heat gain due to solar radiation through epoxy coated glass carried out in this research, is far from complete and could form the basis for further research.

Future work may cover some of the following issues:

1. Measure the daylighting intensity that passes through epoxy coated glass using photometric instruments in order to provide visual comfort by preventing glare and high contrast.

2. Study all the parameters that could affect thermal comfort such as temperature and humidity in the presence of the epoxy coated glass scaled model.

3. Improve the thermal efficiency of epoxy coated glass by applying other layers of the same or other materials; and then retest heat gain through this type of glass.

4. Study the mechanical properties of epoxy coated glass by applying compression, tension and shear forces, and study the durability of this material.

In the field of spreading and wettability of epoxy resin on glass surfaces; future work may cover the following: further investigation of the spreading of cured epoxy resin on glass and glass-like surfaces at low and high temperatures with different curing agent concentrations or with uncured epoxy resin (without any curing agent).
References


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انتشار مادة الإيبوكس على الأسطح الزجاجية

الخليصة

لقد تم اختبار انتشار قطرة صغيرة من مادة الإيبوكس اللاصقة - والتي بلغ حجمه 1.5 ميكرونتر - على سطح زجاجي بهدف تطوير نوع جديد من الزجاج المطلقي بمادة بوليميرية (الإيبوكسي) طاردة للماء، وهذا النوع من الزجاج المطلقي قادر على تقليل كمية الحرارة المكتسبة داخل أي مبنى.

وقد أدت الظاهرة لانتشار وانتشار البلاك ببعض الأطعمة والمضادات الحيوية في العملية الطيران، فقد تم دراسة ديناميكية الانتشار لتركيزات مختلفة من العامل المعالج لمادة الإيبوكس اللاصقة (15:200، 18:200، 21:200) على السطح الزجاجي. وقد أظهرت جميع التركيزات للعامل المعالج لمادة الإيبوكس سلوك جيد للانتشار على الأسطح. في حينما زاد تركز العامل المعالج لمادة الإيبوكس تقل ديناميكية الانتشار على السطح.

ولقد تم اختيار قابلية السطح الزجاجي المطلقي بالبوليمير لطرد الماء من على سطحها، ووجد أن سطح مادة الإيبوكس يملك القدرة على طرد الماء من على سطحها.

لقد زودتنا تقنات الطلاء المغمس بسطح زجاجي مطلقي ببوليمير شفاف المظهر معطياً الزجاج المظهر الطبيعي المعتاد. وهذا يسمح بمرور الأشعة المرئية مزودة المبنى بإضاءة طبيعية.

وكما وجد أن الزجاج المطور المطلقي بالبوليمير - الإيبوكس - يقلل الحرارة الممتدة الناتجة عن الإشعاع الشمسي. وقد استخدم "Kip & Zenon" بيرانونتر مع المجمع الشمسي لقياس الإشعاع الشمسي الممتずっと من قبل السطح الزجاجي. وفقد أظهرت النتيجة أن السطح الزجاجي المطلقي بالإيبوكس زودنا بانخفاض في الحرارة المكتسبة بنسبة ما بين 23% - 60%.
انتشار مادة الإيبوكس على الأسطح الزجاجية

 رسالة مقدمة من الطالبة:

 حنين نمر الدحدوح

 إلى جامعة الإمارات العربية المتحدة

استكمالاً لمتطلبات الحصول على درجة الماجستير في علوم وهندسة المواد

2006 – 2005