

Synthesis, Purification, and Analytical Evaluation of DGEBA Epoxy Resin via Glycidylation of Bisphenol-A



Author of the report: Sonu Kumar

Roll no: 244107123

MTech Chemical Engineering (Petroleum Science and Technology)

Submitted to: Prof. R. Anandalakshmi & Prof. Poonam Kumari

Date of submission: 22/09/2025

Submitted at Department of Chemical Engineering Indian Institute of
Technology Guwahati

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1 Introduction

Epoxy resin is a thermosetting class of polymer having two or more oxirane groups in its structure. which is widely accepted due to exceptional mechanical strength, adhesion, Dimensional stability, chemical corrosion resistance, and resistance to thermal degradation. This material is broadly used in adhesive, coating, electronic, Aerospace material, and structural components across a wide range of industries.(Gonçalves et al., 2022; Zhong et al., 2022) . Additionally, the role of epoxy resin is adhesive and sealant is making them essential in the application of packaging and construction, where a strong bond and durability are required(Dai et al., 2019). The most ordinary Epoxy resin is **diglycidyl ether of bisphenol-A** (Fig.1, DGEBA), which covers over 90% of the epoxy resin market due to its excellent performance and processability(Kalita et al., 2023). The properties of DGBPA are strongly affected by how many repeating units are present in the structure. If the molecular weight is Lower, it is typically in liquid form, while higher molecular weights exhibit higher viscosity or exist as a solid.

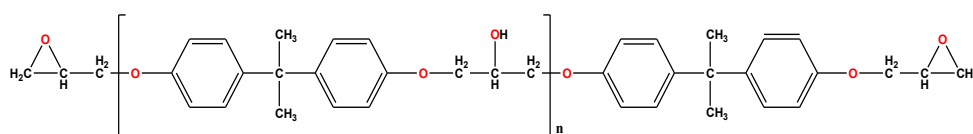


Fig. 1 Chemical Structure of Diglycidyl Ether of Bisphenol-A (DGEBA)

Other similar types of epoxy prepolymer, such as **Triglycidylaminophenol (TGAP)** or **Tetraglycidyl methylenedianiline (TGMDA)**, are extensively used in the aerospace industry, especially for the preparation of aerospace components or those places where high-temperature service is required(Shree Meenakshi & Pradeep Jaya Sudhan, 2016; Vidil et al., 2016) .

Cycloaliphatic oxide 3,4-epoxycyclohexylmethyl-3',4'-Epoxycyclohexane carboxylate (ECC) and **bis(3,4-epoxycyclohexylmethyl) adipate (BECHMA)** are a special type of epoxy resin which is commonly used to enhance UV radiation and resistance to high temperature.

Aluminium conductor composite core (ACCC) materials are mostly used in structural reinforcement in power lines (Tomasi et al., 2016).

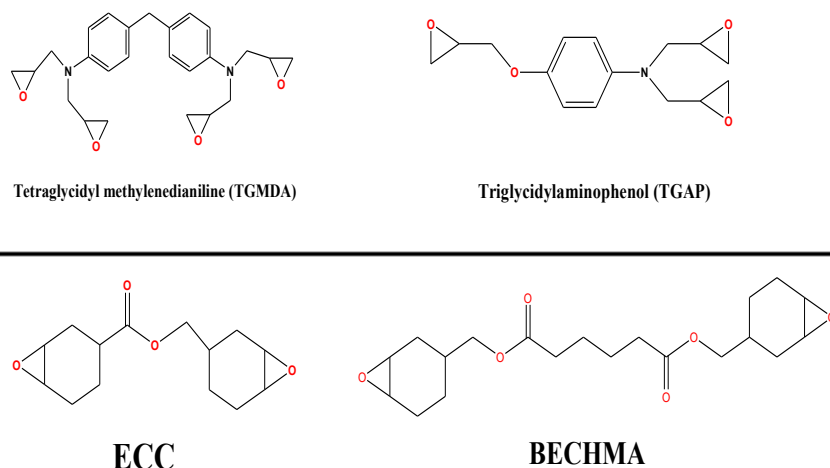


Fig. 2 triglycidylaminophenol (TGAP) and tetraglycidylmethylenedianiline (TGMDA)

The preparation of **DGEBA** (Fig.2) involves a two-step process first step is the **glycidylation reaction**, in this step **Bisphenol-A(BPA)** is reacted with **Epichlorohydrin(ECH)**, and a small amount of phase-transfer catalysts like **Tetrabutylammonium Bromide (TBAB)** are sometimes used to enhance the interaction between the aqueous base and organic reagents, to form a halohydrin as an intermediate. and the subsequent process is **dehydrohalogenation** in the presence of sodium **hydroxide(NaOH)**, resulting in the formation of an epoxy functional group on an aromatic base(C. Chen, 2025; Ma et al., 2016). The overall Reaction gives us a viscous pale-yellow resin with outstanding strength after the curing agent forms a durable thermoset with a crosslink network. Despite all these advantages, **unmodified DGBPA is inherently brittle**, so we need to use modifiers or fillers to increase the toughness or thermal resistance.

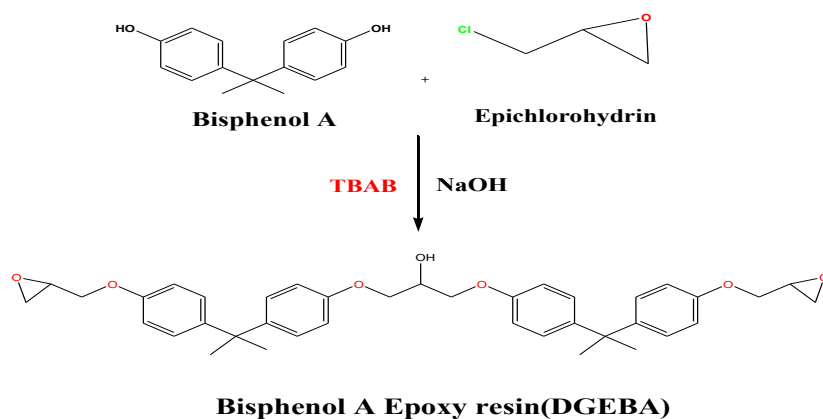


Fig. 3 Synthetic route to bisphenol A epoxy resin (DGEBA).

Bisphenol-A(BPA) is Toxic, an endocrine disrupter, and Epichlorohydrine(ECH) is poisonous, carcinogenic, it is harmful to the environment and health; both are obtained from petroleum feedstocks (Kalita et al., 2023; Liu et al., 2021). Moreover, the non-biodegradability of regular epoxy resin leaves a footprint for long-term as plastic pollution, so urgent need to replace the petroleum-based synthetic Epoxy resin(Saitta et al., 2022). We heavily rely on petroleum feedstock, which is unsustainable due to the depletion of fossil resources and the urgent need to reduce greenhouse gases (Liu et al., 2021).

2 Experimental setup

2.1 Materials

All chemicals used in this study were of reagent grade and employed without further purification to maintain consistency and reliability in the synthesis process. Bisphenol-A (BPA, $\geq 99\%$ purity) and epichlorohydrin (ECH, $\geq 99\%$ purity) were procured from **Himedia Laboratories Pvt. Ltd.** Sodium hydroxide pellets (NaOH, analytical grade) were utilized as the base. Organic solvents, including toluene and isopropanol, were employed to facilitate dissolution, mixing, and purification, while distilled water was used during the washing steps. Anhydrous sodium sulfate (Na_2SO_4 , $\geq 99\%$ purity) was applied as a drying agent, and tetrabutylammonium bromide (TBAB) was used as a phase-transfer catalyst.

For analytical evaluation, pyridine ($\geq 99\%$ purity) and hydrochloric acid (HCl, 37%) were used to prepare pyridinium chloride, which served as the reagent for determining the epoxy equivalent weight (EEW). Phenolphthalein indicator serves as an indicating agent that the titration is completed or not.

2.2 Method

A magnetic stirrer with a heating plate was employed, and a 2000 mL beaker filled with water was placed on top of the stirrer to serve as a constant-temperature bath. The temperature of the bath was maintained between 60–70 °C. A three-neck round-bottom flask was placed inside the beaker. The central neck was fitted with an overhead reflux condenser in a vertical orientation, and the joint was secured tightly using either a Keck clip or a greased joint to ensure proper sealing. The condenser was operated with a moderate water flow rate, supplied from the inlet at the bottom to the outlet at the top, to prevent excessive pressure that could damage the condenser. The flow direction was carefully maintained to avoid reversal, which could cause steam buildup. (Although tap water was used as the coolant, liquid nitrogen may also serve as an alternative cooling medium.)

The reflux condenser was essential to prevent the loss of volatile epichlorohydrin (boiling point ~ 117 °C) and to avoid pressure buildup inside the reaction flask, thereby ensuring an open reflux system. The second neck of the flask was equipped with a thermometer for monitoring the internal reaction temperature, while the third neck was sealed with a rubber stopper. An additional thermometer was placed in the beaker to measure the bath temperature. The reaction mixture was charged with **Bisphenol-A** (BPA) (45.6 g, 0.2 mol; molecular formula $C_{15}H_{16}O_2$), used in its white crystalline solid form as the core monomer, and **Epichlorohydrin** (ECH) (62.7 mL, 0.8 mol, excess; molecular formula (C_3H_5ClO)), supplied as a liquid, which acted as the glycidylating agent. At specific stages of the reaction, **Tetrabutylammonium bromide** (TBAB, 0.1 g) was introduced as a phase-transfer catalyst to enhance reaction efficiency.

The magnetic stirrer was switched on, and the stirring speed was set to 500 rpm. The bath temperature was maintained at 60–70 °C until the reactants were completely dissolved (~ 45 –50 min), at which point the solution appeared crystal clear.

A **sodium hydroxide** (NaOH) solution (0.4 mol, 50% w/w) was employed as the base catalyst for the glycidylation reaction. The solution was introduced through a dropping funnel attached

to the third neck of the round-bottom flask, added dropwise at a rate of approximately one drop per second, over a period of 1–2 hours. Upon completion of the addition, the reaction mixture turned milky white. Subsequently, 100 mL of toluene was added to a round 3-neck bottom flask to dissolve the epoxy resin.

The toluene–epoxy mixture was then transferred to a separating funnel to remove residual sodium hydroxide and the byproduct sodium chloride. Hot Deionized water (50 mL, 60–80 °C) was added for washing. If the total volume exceeded 50% of the separating funnel’s capacity, the mixture was divided into batches. The separating funnel was closed with a rubber stopper, shaken vigorously for 10–20 seconds, and vented frequently to release pressure. The funnel was then secured on a stand and left undisturbed for ~20 minutes to allow phase separation. The lower organic phase (toluene + diglycidyl ether of bisphenol-A, DGEBA) separated from the upper aqueous phase (water + salts) due to its lower density. The aqueous layer was drained from the bottom, while the organic phase was retained. The washing process with hot water was repeated 3–4 times to ensure thorough removal of residual salts and base.

The organic phase, consisting of toluene and epoxy resin, contained trace amounts of water, which were removed using anhydrous sodium sulfate (Na_2SO_4) as a drying agent during the purification step. An appropriate amount of Na_2SO_4 (~5–10% of the organic phase volume) was gradually added to the solution while gently swirling or stirring with a magnetic stirrer (vigorous stirring was avoided to prevent possible resin degradation).

The drying process was monitored by observing the behavior of Na_2SO_4 : clumping indicated water absorption, whereas free-flowing Na_2SO_4 suggested the presence of residual moisture, in which case an additional (1–2 g) was introduced. Once clumping ceased, the organic phase was considered sufficiently dry. For conditions requiring stronger dehydration, magnesium sulfate (MgSO_4) or calcium chloride (CaCl_2) could be employed as alternative drying agents.

It should be noted that Na_2SO_4 is a non-regenerative drying agent and was discarded after use. For large-scale drying, azeotropic distillation of the toluene–water mixture may be considered as an efficient alternative.

Following drying, the DGEBA–toluene solution was separated from anhydrous Na_2SO_4 by vacuum filtration. A filter paper (Whatman) was placed inside a funnel and pre-wetted with a few drops of toluene to ensure proper adhesion to the filter plate. The funnel was then attached to a vacuum flask connected to a vacuum pump via a rubber tube. Upon switching on the pump, the DGEBA–toluene solution containing Na_2SO_4 was slowly poured into the funnel, using a glass rod to guide the liquid and avoid spillage. The vacuum assisted in drawing the liquid

through the filter paper, while Na_2SO_4 crystals were retained on the filter. The drying agent was rinsed with an additional 5–10 mL of fresh toluene to recover any trapped resin.

The filtrate was transferred into a round-bottom flask, which was securely clamped to a stand and attached to a rotary evaporator. The bath temperature was set to 50–60 °C, the pressure adjusted to 70–80 mbar, and the rotation speed maintained at ~140 rpm. The mixture was subjected to rotary evaporation for 60–90 minutes to efficiently remove toluene and obtain purified DGEBA resin.

3 Characterization

3.1 Fourier Transformed Infrared Spectroscopy (FTIR)

Fourier Transform Infrared (FTIR) Spectroscopy is a widely used technique to confirm molecular structure, specifically to identify the functional groups present in epoxy resins. FTIR analysis was performed in the spectral range of 400–4000 cm^{-1} .

Prior to analysis, the ATR (Attenuated Total Reflectance) crystal, composed of diamond, zinc selenide (ZnSe), or Germanium (Ge), was cleaned with acetone. A background scan was first recorded to eliminate unwanted IR absorptions from environmental sources (e.g., atmospheric CO_2 and H_2O) and instrument noise, which could otherwise interfere with the resin spectrum. Subsequently, a small drop of the epoxy resin sample was placed on the ATR crystal, ensuring complete coverage without air bubbles, and the sample scan was recorded. The most significant functional group in this compound is the epoxy (oxirane) ring. The absorption band deformation of the oxirane ring, which exhibits a characteristic absorption peak at **915 cm^{-1}** due to stretching the three-membered oxirane ring, is a sign of an unreacted epoxy group presence of the prepared Resin(Shree Meenakshi & Pradeep Jaya Sudhan, 2016) . The medium intensity of the peak, as shown in (Fig. 4), indicates a relatively low concentration of epoxy groups in the sample.

In the FTIR analysis, the Y-axis represents the % Transmittance, which simply means how much IR light passes through the sample at a given wavenumber(cm^{-1}), represented on the X-axis. The **Transmittance is higher** when the sample absorbs a minimal amount of IR energy, allowing most of the IR light to pass through, and thus no peak is formed at that frequency. This indicates **a very small amount of that functional group is present** in the sample. If the **Transmittance is Low**, it means that it absorbs more IR light and forms a peak at a particular Frequency. It represents that the **functional group is present in that sample in a very high amount**.

For the FTIR analysis, the experiment was conducted in the Analytical Laboratory of the Chemical Engineering Department. The FTIR instrument was first switched on by connecting the power supply and opening the sample compartment cover. Prior to sample loading, the diamond ATR crystal was cleaned with ethanol or acetone to ensure the removal of any contaminants. The operating software(Spectrum IR) was then opened, and a background scan was performed to eliminate contributions from residual impurities on the crystal or atmospheric interferences (e.g., CO₂, H₂O vapor).

The epoxy resin sample, being in liquid form, was directly placed on top of the ATR crystal without applying additional gauge pressure (20–30 psi is typically required for solid samples to ensure proper contact). The scan was then initiated, and the resulting FTIR spectrum was obtained. The spectral data (wavenumber vs. transmittance) were exported as a CSV file for further analysis.

Data processing was carried out using Origin software, where the FTIR spectrum was plotted and analyzed for functional group identification. In the spectrum, sharp or intense peaks correspond to lower transmittance values at specific wavenumbers, indicating strong absorption by characteristic functional groups. Conversely, weak or shallow peaks suggest the presence of the corresponding functional groups in lower concentrations within the sample.

The FTIR spectrum of DGEBA is shown in **Fig.4**. The Broad peak of **-OH** (hydroxyl group) that appears at **3400 cm⁻¹** confirms the presence of some water or alcohol like groups present, or may be due to the possibility of forming a higher molecular weight oligomer of the tetrafunctional epoxy resin(Shih & Ma, 1998). The peaks corresponding to the **-CH₂** group of DGEBA appear at **2900 cm⁻¹** (Shree Meenakshi & Pradeep Jaya Sudhan, 2016) .The peak around **465 cm⁻¹ - 572 cm⁻¹** tells us about heavy atoms like halogens (chlorine **C-Cl**, bromine **C-Br**). The wavenumber range of **695 cm⁻¹ to 828 cm⁻¹** represents the bending vibration of the **C-H** bond. The characteristic peaks that appear at **1034 cm⁻¹ - 1128 cm⁻¹** confirm the presence of a strong ether bond(**C-O-C**), which is common in epoxy resins, and **1243 cm⁻¹** represents **C-O** bond stretching in the aromatic ring (Meadows et al., 2018) . **1508 cm⁻¹** and **1606 cm⁻¹** represent the aromatic **C = C** stretching confirm Benzene ring vibration(Park & Jin, 2005) . Overall spectrum confirms that the sample is Epoxy resin based on Bisphenol-A(DGEBA) with different functional groups like Ether linkage, Hydroxyl group, Epoxy ring, Aromatic ring, and some Halogens.

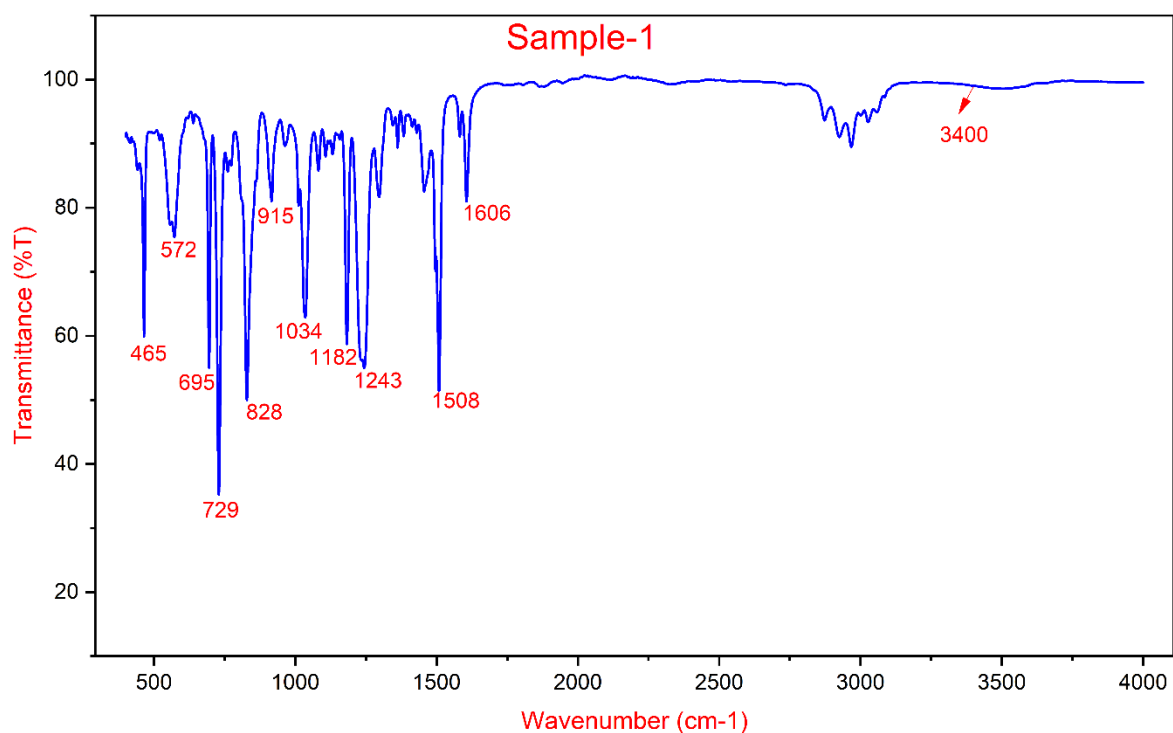


Fig. 4 FTIR spectroscopy of DGEBA

First, we tested the commercial epoxy resin sample obtained from the market, which exhibited a characteristic peak at 915 cm^{-1} with a transmittance of 71.31%. Subsequently, all laboratory-prepared samples were analyzed, and their spectra were compared. In Sample-1, the transmittance was 81.03%; in Sample-2, 76.29%; in Sample-3, 78.03%; and in Sample-4, 75.14%. Among the four prepared samples, the transmittance values were relatively close to those of Sample-4, indicating that the peak intensity in Sample-4 was higher than in the other samples. A comparative analysis is presented in the figure provided in the report. Based on this analysis, it can be confirmed that the materials synthesized in the laboratory were epoxy resins.

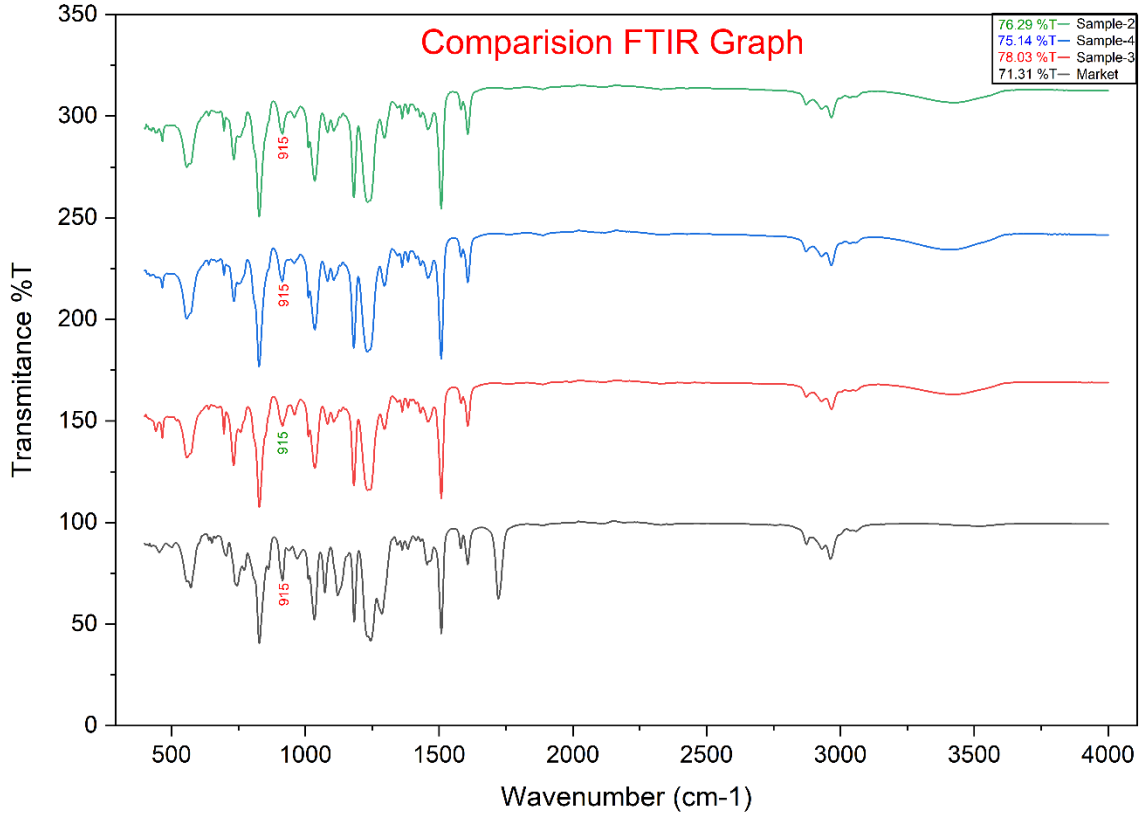


Fig. 5 Comparison of FTIR spectroscopy of different samples

3.2 Measurement of Epoxy Resin Viscosity

Viscosity(η) is a significant Rheological property of the Epoxy resin System. It is typically expressed in SI units of **Pascal-seconds (Pa·s)** or **milliPascal-seconds (mPa·s)** for a viscosity conversion factor of **1cP** is **(0.001 Pa · s and 1mPa · s)**. It defines how the behaviours of flow of epoxy, which play a critical role in its application to curing, processing, and mechanical performance. It describes the resistance of a liquid to deformation under shear stress and quantifies how easily the resin flows during processing. In Newtonian Fluid (water), viscosity remains constant with shear rate, but for non-Newtonian Fluid(like Epoxy), viscosity can change with shear rate (Y. Chen et al., 2024) .

Factors Affecting the Epoxy Resin, the first is **Temperature**: viscosity decreases with an increase in temperature due to enhanced molecular mobility, and 2nd **Concentration**: with an increase in the concentration of resin, viscosity increases due to enlargement of the polymer chain, and increased intermolecular interaction (Bekhta et al., 2019) . **Additives and Fillers**: The addition of nanofillers (such as graphene nanoplatelets or minerals) in the Epoxy resin

increases the viscosity, which affects the mechanical strength (Li et al., 2017). Commercial Liquid Epoxy resin Viscosity is approx 11000 cP-14000 cP (11-14 Pa.s).

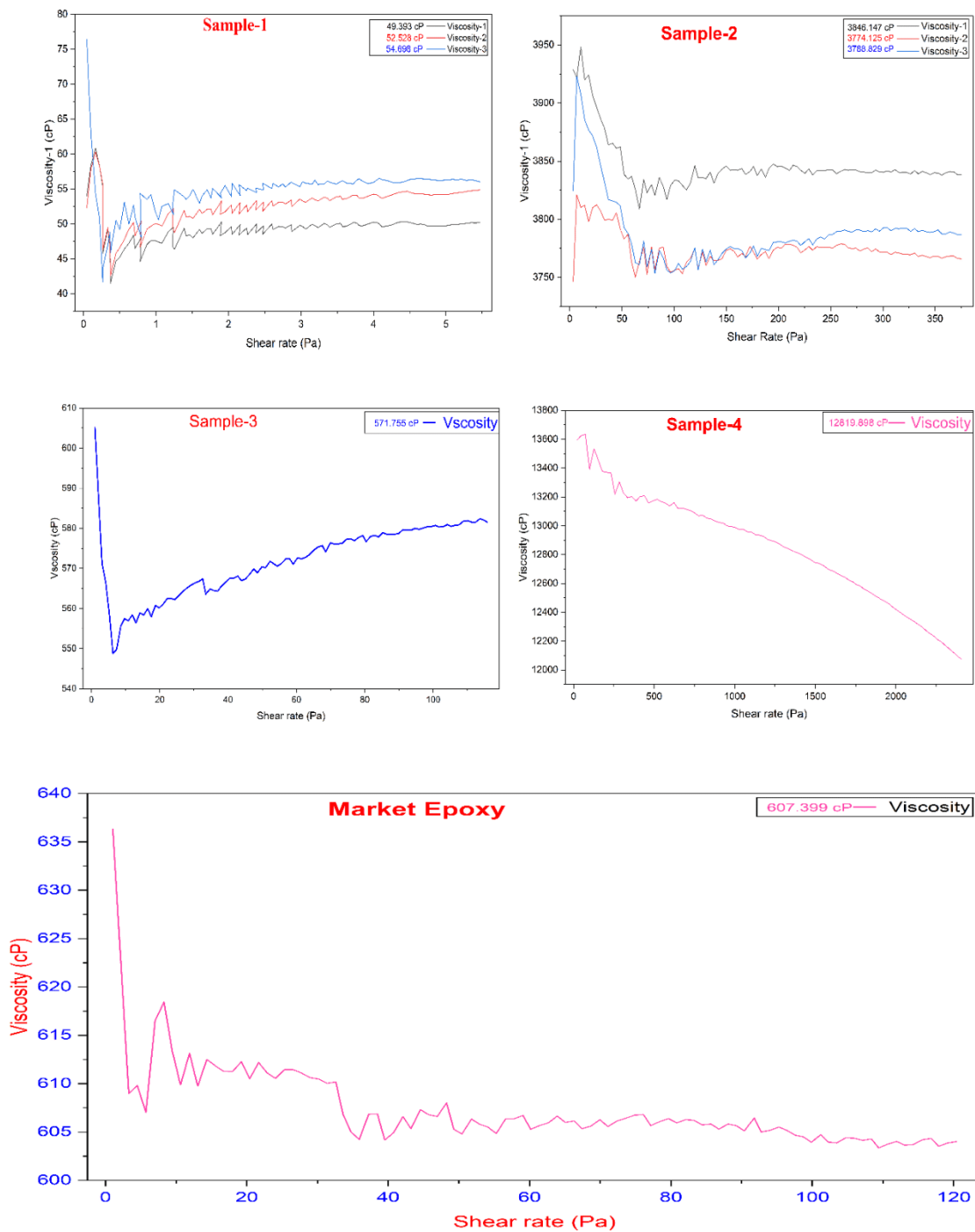


Fig. 6 Viscosity Graph of all the samples and Market Epoxy Resin

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