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Preparation and Characterization of Polymeric Adhesives for Concrete Applications

A Thesis
Submitted to the Council of the College of Materials Engineering/ University of Babylon in Partial Fulfillment of the Requirements for the Master Degree in Materials Engineering/ Polymer

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بِسْمِ ٱللَّهِ ٱلرَّحْمَٰنِ ٱلرَّحِيمِ

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لِنُورِهِۦ مَن يَشَآءُ ۚ وَيَضْرِبُ ٱللَّهُ ٱلْأَمْثَٰلَ لِلنَّاسِ ۗ وَٱللَّهُ بِكُلِّ شَىْءٍ
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سورة النور الآية 35

Supervisors Certification

We certify that this thesis entitled (**Preparation and Characterization of Polymeric Adhesives for Concrete Applications**) was prepared by (Zainab Majid Mohammed) under our supervision at University Babylon/College of Materials Engineering/Department of polymer and petrochemical industries, in Partial Fulfillment of the Requirements for the Award Master Degree of Science in Materials Engineering / Polymers Engineering.

Signature:

Name: Prof. Zoalfokkar Kareem Mezaal Al-obad

(Supervisor)

Date: / / 2022

Dedication

I am writing this letter and I would like to dedicate it to my father, my God have mercy on him, and make him live in peace.

To whom you offered my happiness and comfort over her happiness, Mom

To my brothers;

Mustafa
Mortada
Maher
Ahmed

To my sister;

Zahraa

To my husband;

My soul mate (Ali)

With Respect

ZainabMajid

Acknowledgment

I would like to thank ***Allah*** who gave me the ability and desiret complete this work.

I would like to express my deepest sense of gratitude to my supervisors ***Prof. Zoalfokkar Kareem Mezaal Al-obad*** for his continuous advice and encouragement throughout the course of this work. I thank his for the guidance and great effort his made in supervising this scientific field.

Additionally, I would like to thank the sisters that life gave birth to their ***Rusul Mohammed*** and ***Sura Kamel*** for standing by me and supporting me, without him I would not have been able to complete this work.

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Finally, I would like to express my appreciation and love to the members of my family and my friends for their support inspiration and care during this work.

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Abstract

The world is currently interested in preserving the environment and reducing pollution through the use of recycled waste, as well as reducing costs. The strict environmental laws and lack of dumping sites in urban areas are making the disposal of demolition wastes problematic. As concrete waste is a dangerous pollutant to the environment.

Recycling concrete is therefore an important research topic in order to prevent global warming. Building materials such as cement and concrete waste increase global warming, and it is a global problem at the present time. For these reasons, there is an urgent need to remove waste concrete from the environment. where portland cement (C) is added once and waste concrete (W) again at an added percentage of (0,3,6,9, 12,15and20wt.) to Polysulfide Rubber (PSR) taking into account the Considering that the adhesive retains the adhesion and tensile properties as a basic thing parallel to its importance in reducing the cost.The composite materials were prepared by mixing polysulfide Rubber (with a ratio of 95% polymers: 5% hardener) with cement once and waste concrete again.

Fourier Transform Infrared Spectrometer (FTIR) results show that there is no chemical interaction between polysulfide Rubber samples and cement once or waste concrete.The results of the tensile test show that the ultimate tensile strength increases for polysulfide compound with the increase of the waste concrete content ,and it is higher than ultimate tensile strength of the pure polysulfide samples but when cement is added to polysulfide, the ultimate tensile strength of cement reinforced polysulfide increases slightly even the addition percentage (12%) (0.199 MPa) at the level of the ultimate tensile strength value of pure polysulfide samples then the decreases tensile strength at the percentage (15% and 20%).The results of the elongation show that the elongation of the composite materials decreases with the increase in

the amount of addition of cement and waste concrete and the decrease is more for samples reinforced with waste concrete than reinforced with cement.The pull off adhesion test results show that the adhesion strength show improvement when adding from 3% to 20% of the cement and waste concrete.The results of Shore (A) hardness test show that the values of reinforced polysulfide are higher than that of pure polysulfide Rubber. Scanning Electron Microscopy (SEM) images show agglomeration in waste concrete and a rough and irregular surface which is reflected on the mechanical properties. It can be seen that the shape of waste concrete is more rough and larger than cement and the cement particles have a finer shape than waste concrete.

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List of Abbreviations

Abbreviate	Meaning
AEC	Air entraining cement
Al_2O_3	Aluminium oxide
ASTM	American Society for testing materials
BFSC	Blast furnace slag cement
C	Cement
$CaCO_3$	Calcium carbonate
CaO	Calcium oxide
CC	coloured cement
CO_2	Carbon dioxide
EP	Epoxy
E_{coh}	the amount of energy required to separate molecules to an infinite distance
FTIR	Fourier transform infrared
HAC	High alumina cement
HpC	Hydrophobic cement
IR	Infrared rays
LED	Light emitting diode
LEP	Large Electron Positron
LHC	Low heat cement
OPC	Is the interoperability standard for the secure and reliable exchange of data in the industrial automation space and in other industries
PC	Polycarbonate
PE	Polyethylene

PMMA	Poly methyl methacrylate
PP	Polypropylene
PSR	Polysulfide Rubber
PTFE	Poly tetra fluoro ethylene
PVC	Polly Vinyl Chloride
PzC	pozzolanic cement
QSC	Quick-setting cement
R	Alkyl groups
RHC	Rapid hardening cement
SEM	Scanning electron microscope
SiO_2	Silicon dioxide
SRC	Sulphate resisting cement
Thiokol LP-2	Liquid Polysulfide Polymers
TiO_2	Titanium dioxide
UV	Ultraviolet
VOC	Volatile organic compound
V	the molar volume
W	Concrete waste
WC	white cement
δ	the solubility parameter

Chapter One

Introduction

1.1 General Introduction

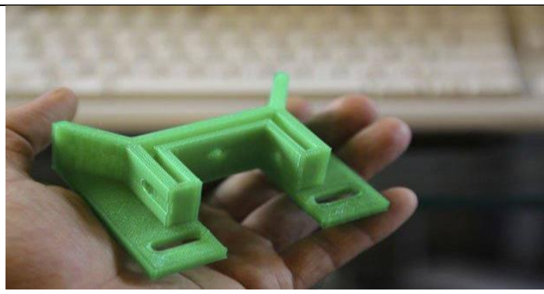
Considerable efforts and advances had been made over the recent years to develop specialty polymers that suit particular applications and satisfy the construction market demand. The world is currently interested in preserving the environment and reducing pollution through the use of recycled waste, as well as reducing costs[1].

In general, polymers are very important and have wide applications in industry for example, they are used in packaging, damping, vibration, insulation, resale food storage bags, and pipe, production of plastic bottles and medical devices….etc[2]. Adhesives are one of the very important polymers. Adhesives include different types, some of which are used in concrete, polymer and metal applications .Concrete adhesives have wide used, including bridge expansion joints such as polysulfide adhesives[3].

Recycled materials are used to reduce costs.Waste materials in polymers such as waste concrete and Portland cement can be used to strengthen polymers in general and adhesives in particular. As concrete waste is a dangerous pollutant to the environment[4].

Limited attempts were made to develop specialty polysulfide possessing improved tensile and bond properties resulting from inter-grinding waste concrete[5].

Polysulfide Rubber (PSR) is flexible, chemically resistant elastomers with a linear polymer structure consisting of aliphatic carbon functional groups separated by one or more sulfur atoms The polysulfide use as shown in figure (1.1). PSR sealants are excellent adhesives and adhere to a wide variety of surfaces but price inevitably plays a major role in the final selection of material used for high volume applications[6].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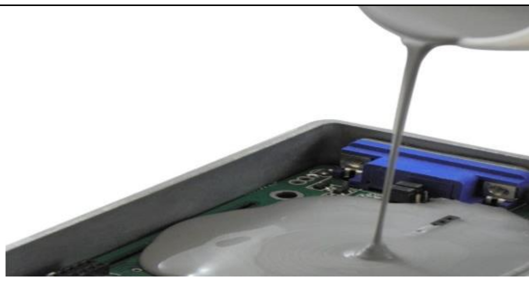
	
(C)	(d)

Figure (1.1): Photos of polysulfide resins used in (a) dental impression compounds ,(b) cast printing rolls ,(c) casting compounds for flexible molds, and (d) electrical potting[6].

On the other hand, waste concrete attracted enormous attention to environmental preservation is an area of civil engineering that could be supported by the use of recycled concrete, which: minimizes the discharge of solid residues that pollute the environment, reuses materials that are considered waste and that do not have a significant cost [7 and 8].

There are over ten different types of cements that are used in construction purposes, and they differ by their composition and are manufactured for different uses including rapid hardening cement (RHC), quick-setting cement (QSC), low heat cement (LHC), sulphate resisting cement (SRC), blast furnace slag cement (BFSC), high alumina cement (HAC), white cement (WC), coloured cement (CC), pozzolanic cement (PzC), air-entraining cement (AEC), and hydrophobic cement (HpC)[9].

1.2 Adhesives for Industrial Applications

Adhesives are designed for specific applications. Besides their role in the adhesion process, they can be used for other purposes, such as sealing agents, in order to eliminate the effect of self-loosening caused by dynamic loads, sealing of areas to prevent oxidation and corrosion, waterproofing, etc. Sealants can be used as electrical or thermal insulators, fire barriers and products for smoothing, filleting or flying. The materials that are used as sealants have lower strength than those used as adhesives because sealant formulations contain large amounts of inert filler material for cost reduction and gap filling purposes. Certain sealants, like adhesives, can be used to assemble parts, and many adhesives can be used to seal. The adhesives and sealants are mainly used to bond the following substrates: metals, plastics (thermosets and thermoplastics), composites, foams, elastomers, wood and wood products, glass and ceramics and sandwich and honeycomb structures [10-13].

1.3 Aim of The Current Work

This work aims to prepare low cost adhesives using waste concrete and cement particles. In current work, PSR ,PSR/W and PSR/C sheets were prepared using cement and waste concrete. To improve structural, morphological and mechanical properties were studied by FTIR, SEM, tensile, pull off and hardness properties.

1.4 Objectives

1-Take polysulfide adhesive.

2-preparation waste concrete by:

A-concrete cleaning

B-Drying out concrete

C-Grinding

D-Sieving

3-Adding concrete waste to polysulfide

4-Manufacture a composite material

5- Testing

A-Tensile Test

B- Hardness Test (Shore A)

C- FTIR

D-Pull-Off Adhesion testing

E- SEM

Chapter Two

Theoretical Part

&

Literatures Review

2.1 General Introduction

Adhesive bonding is the most versatile of all joining techniques and can be used to join plastic parts to each other or to other materials such as metals, ceramics or wood. A range of joint strengths is available, ranging from low strength putty and caulking compounds, which are used only for space and void filling to high strength structural adhesives used in the automotive and aerospace industries. In simplest terms an adhesive is applied to the substrate or adherend, surfaces the joint is formed by holding the components together while the adhesive cures/hardens to develop structural properties, forming a bond to both surfaces[7].

2.2 Adhesives

Adhesives can be used to join different types of plastics and composites to themselves and to other materials, such as metals, wood, fabric, film, or cardboard. With a clean substrate surface, most materials can be adhesively bonded to a similar or dissimilar material; for difficult materials (e.g., those with a low surface energy such as PE, PP, or PTFE), primers, or special surface preparation techniques can increase bond ability significantly. Substrates can take any form including blocks, films, sheets, particles, sandwich structures, or honeycomb cores. Applications where polymers are joined using adhesives are incredibly varied and include the following:

• *Cosmetic Containers*: many attractive polymeric materials can be used to great effect when joined to form receptacles and similar items, adhesive bonding is often the most effective, economical and aesthetic way to achieve.

• *Lighting*: adhesives are often used to manufacture plastic lighting structures, including low power systems (e.g., LED, LEP, and electroluminescent systems).

• *Optical Components*: the increasing use of high refractive index polymers (PMMA, PC, and nylon) in items such as glasses, cameras, photonics, etc., has necessitated the development of a range of ultraviolet (UV) curing adhesives to enable the joining of such materials so that movement and misalignment are minimized.

• *Sporting Goods*: virtually all items used for sport will contain some level of adhesive bonding, whether it can be to bond the rubber surface of a table tennis bat to the highly complex laminated construction required by skis and associated boards (surf, snow, kite, sail, etc.). Many high quality bicycles (mountain and road) are constructed from a mixture of metal and polymer composite materials which can be joined only using adhesive technology.

• *Office Equipment*: adhesives are used within office electronics, for example, to attach and retain components on circuit boards, and to attach fabric on furniture.

• *Toys*: whether constructed of natural polymeric materials (e.g., wood and leather) or synthetic thermoset/thermoplastics, adhesive bonding forms an intrinsic method of assembly, in particular due to its ability to join dissimilar materials and avoid the use of fasteners, which can present a safety risk should a component become detached.

Structural adhesives are used in the aerospace, automotive, sport, and appliance industries for joining structural components. Adhesives are used in assembling medical components, such as orthopedic braces, hearing aids, transducers, infusion devices and pumps, medical electronic equipment, catheters, and attaching stainless steel hypodermic needles to plastic housings. Adhesive applications also include thread locking, thread sealing, retaining, and gasketing[14 and15].

Automotive applications include bonding two-piece thermoplastic bumpers and rearview mirrors bonded to glass windshields. In addition, the attachment of trim is almost entirely done using adhesives ranging from double-sided structural tape through to polyurethanes and cyanoacrylates. The benefits of using adhesives include the avoidance of producing holes, more rapid assembly, tolerance compensation and aesthetic benefits[11,15-16].

The joining of polymer composite materials (continuous fiber reinforced materials and honeycomb sandwich structures) has always presented challenges to the engineers. For though these materials offer significant advantages over conventional materials in terms of weight saving, performance, and durability, their inherent structure (anisotropic laminate) makes the joining of these materials to others difficult. Although mechanical fastening is used, the anisotropic laminate structure is sensitive to any form of defect or damage such as holes, resulting in local stressed areas and a degradation in the overall performance. The most favorable approach therefore is to employ adhesive bonding, where the load can be evenly spread over a large area. There are many examples where this has been applied with great success, including bonding of composite pipe work, the joining of ship hulls (steel) to the upper composite superstructure, and in the joining of sandwich structures for the automotive and aerospace industry. In all of these examples, different approaches in terms of joint design, material/process selection, and assembly mode have been taken to provide an effective bonding solution in each case [11].

Since the 1950s, adhesive joints have been used increasingly in shoe manufacturing as an alternative to sewing and other fastening techniques, indeed within the leisure sector (trainers, etc.), joining is almost exclusively adhesive-based. The primary materials used include leather, canvas, textiles, nylon, and PVC for the uppers, and leather, rubber, EVA, PVC and polyurethanes for the soles. Depending on the material combination and the area of shoe to be joined ,a number of adhesive types are used, the majority based on rubbers, hot-melts, or polyurethanes. More detail is provided in Table (2.1) [11].

Table 2.1: Adhesives Used in Shoe Bonding[7].

Shoe Joining Area	Adhesive
Mounting	Cement (rubber adhesive)
Heel covering	Polyurethane
Heel attachment to sole	Polyester hot-melt
Box toe bonding	Polyamide hot-melt
Shank and cushion bonding	Waterborne polyurethane
Lift attachment	Polyester hot-melt
Sticking of socks lining	Polychloroprene
Upper to sole bonding	Polyurethane, polychloroprene

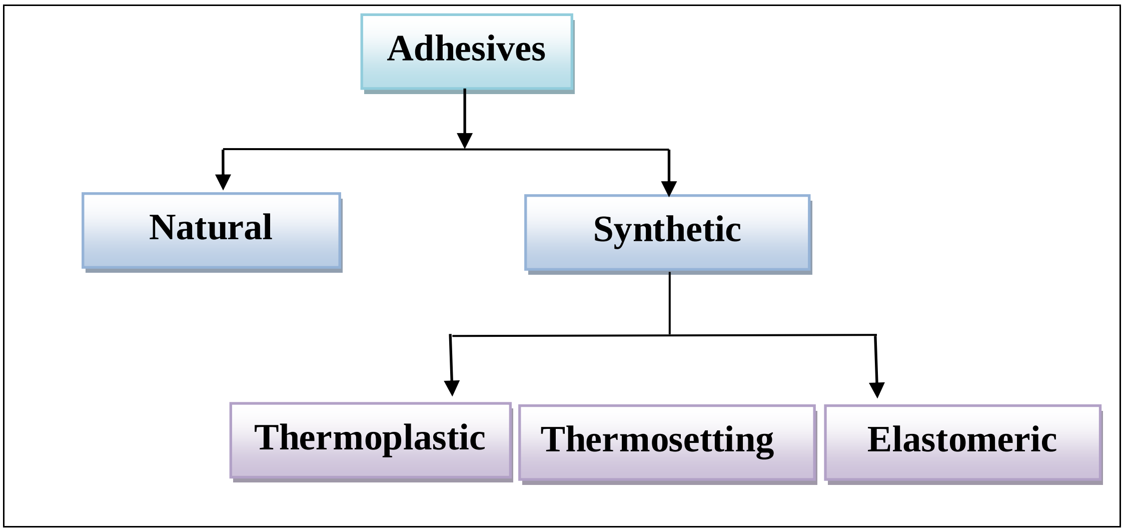
• *Adhesives for Building Construction:* Glues and sealants have been used in construction since Biblical times; but the synthetic adhesives achieved prominence only after World War II. Even in recent decades, however, changes have been dictated by new adhesive materials and new building materials. Today, many types of adhesives are in use throughout industry, including the construction industry. Some adhesives require sophisticated application techniques while others can be put in place by trowel, brush, spray, or spot application because most construction applications are carried out at the job site[17].

Adhesives are often used in construction, as are concrete and ceramics. One of these adhesives materials used in construction is epoxy. Epoxy resin concrete has high strength, good durability, fast growth strength, good toughness, forming time is short, and the advantages of easy construction, etc. As a high performance material, it is applied in many fields such as machinery, construction, chemical industry….. etc. Although EP resin concrete has been widely used in civil engineering in recent years, most of them are used as patching materials, rather than being made into prefabricated components that can be used in engineering to improve structural performance. With the continuous improvement of domestic production technology and the rapid development of the construction industry, China has begun to study and explore more economical and environment-friendly materials with the advanced technology of foreign countries. Based on epoxy resin concrete component of truss structures, with its high strength, better seismic resistance than concrete structure, lower cost than steel structure, better plasticity, assembly and simple construction technology than wood structure, it has a very broad prospects for development and application space[18].For more information about types of adhesives and their application see (Appindex B).

2.3 Classification of Adhesives

This section presents classifications of adhesives from a number of points of view including function, source, physical form, mode of application and setting, chemical composition, Society of Manufacturing system, Rayner system, and others. A simple classification is depicted in

Figure (2.1) . Adhesives are either produced from a natural source such as starch glue or, as is the case with the majority of consumptions, they are synthesized from basic hydrocarbons. The synthetic group consists of thermoplastic and thermosetting adhesives, both of which follow the definitions used in plastics for thermoplastic and thermosetting polymers[19].



Figure(2.1):A simple classification of adhesives.

2.3.1 Natural Adhesives

This term is used to include vegetable- and animal-based adhesives and natural gums. These include organic materials such as casein, blood, albumin, hide, bone, fish, starch, resin, shellac, asphalt, chitosan, and inorganic adhesives like sodium silicate. Their use, except for the inorganic adhesives, is mostly limited to paper, paperboard, foil, and light wood. They are in expensive, easy to apply, and have a long shelf life. These adhesives develop tack quickly, but have low strength properties. Most are water-soluble and use water as a solvent. They are supplied as liquids or dry powders to be mixed with water. Some are dispersions in organic solvents[20].

2.3.2 Synthetic Adhesives

This term is usually used to apply to all adhesives other than natural adhesives(i.e., elastomeric, thermoplastic, thermosetting, and alloys). All structural adhesives are synthetic[19].

2.3.2.1 Thermosetting Adhesives

These are materials that cannot be heated and melted after the initial cure. Curing takes place by chemical reactions at room temperature or at an elevated temperature, depending on the type of adhesive. Some thermosetting adhesives require considerable pressure, while others require only contact pressure. Solvents are sometimes added to facilitate application. These adhesives are usually available as solvent-free liquids, pastes, and solids[21]. Thermosetting adhesives are provided as one- and two-part systems. The one part systems usually require elevated temperature cure and have a limited shelf life. The two-part systems have longer shelf lives and can usually be cured slowly at room temperature, or somewhat faster at moderately higher temperatures. A disadvantage is their need for careful metering and mixing to make sure that the prescribed proportions are blended and that the resultant mixture is homogeneous. Once the adhesive is mixed, the useful life is limited [21].

2.3.2.2 Thermoplastic Adhesives

These materials do not cross-link during cure and they can be melted without significant change in their properties. They are single-component systems that harden upon cooling from a melt state, or by evaporation of a solvent or water vehicle. Wood glues are thermoplastic emulsions that are common household items. They harden by evaporation of water from an emulsion. Thermoplastic adhesives are not ordinarily recommended for use at above 66 $^{\circ}$C, although they can be used up to 90 $^{\circ}$C in some applications. These materials have poor creep resistance and fair peel strength. They are used mostly in stressed joints and designs with caps, overlaps, and stiffeners. The materials most commonly bonded are non-metallic material, especially wood, leather, plastics, and paper [20 and 21].

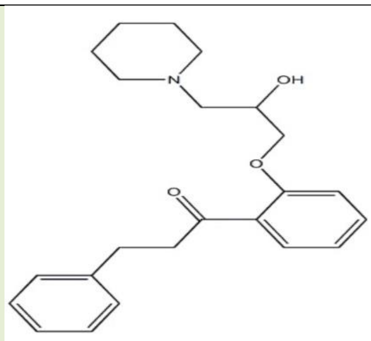
2.3.2.3 Elastomeric Adhesives

These materials are based on synthetic or naturally occurring polymers. They have superior toughness and elongation. Elastomeric adhesives maybe supplied as solutions in organic solvents, latex cements, dispersions, pressure-sensitive tapes, and single- or multiple part solvent free liquids or pastes. Curing varies, depending on the type and the form of adhesive. These adhesives can be formulated for a wide variety of applications, but they are generally used for their high degree of flexibility and superior peel strength. [20 and 21]Some elastomeric adhesives are supplied in film form. Most of these adhesives are solvent dispersions of water emulsions. Temperature environments up to 66 – 204 °C are practical. Elastomeric adhesives never melt completely. Bond strengths are relatively low, but flexibility is excellent. These adhesives are used in unstressed joints on lightweight materials, so they cannot be considered structural adhesives. They are particularly advantageous for joints in flexure. Most of these adhesives are modified with synthetic resins for bonding rubber, fabric, foil, paper, leather, and plastic films. They are also applied as tapes[19].

2.4 Polysulfides (Thiokols)

Polysulfides are flexible materials belonging to the synthetic rubber family. Some of the more important characteristics of polysulfide adhesive/sealants are tabulated. Although polysulfides are primarily used as sealants for automotive, construction, and marine uses, they are used to some extent as flexibilizing hardeners for epoxy adhesives. Their sulfur linkages combine good strength with the ability to rotate freely, resulting in a strong, flexible polymer. Polysulfides utilize atmospheric moisture to accelerate cure. A two-component system is usually used, consisting of formulated polysulfide and formulated lead dioxide catalyst. Moisture converts a portion of the lead dioxide catalyst to a faster-reacting form [22]. Polysulfides cure at room temperature and reach maximum strength in 3 –7 days. Polysulfides and epoxies are mutually soluble in all proportions. Polysulfides are also alloyed with phenolics [21]. Curing agents may be furnished in powder, paste, or liquid form. The activity of the metallic curing agents is a function of surface area, thus increasing the importance of particle size. As it is necessary to obtain a fairly complete dispersion throughout the polymer in order to achieve complete cure, it is generally more effective to combine the lead oxide with a plasticizing agent to form a paste[23 and 24].

The term polysulfide polymers referred at one time exclusively to the high-sulfur-containing polymers as manufactured by the Thiokol Chemical Corp. From 1928 to 1960 they were the only high sulfur polymers available. The solid poly sulfide polymers contained 37-82 % bound sulfur, while the liquid polymers contain approximately 37 %, which gives them their unique chemical properties. Between 1960 and 1976 several new mercaptan terminated polymers having varied polymer backbones were introduced. These are covered in the section entitled "Other Mercaptan Terminated Polymers." The chemical resistance of these various polymers can vary depending upon their backbone structure and on this basis, should be evaluated on their own relative merits[17].



Figure(2.2): Structure of polysulfide Rubber (PSR)[19].

Figure(2.3):polysulfide Rubber (PSR)[19].

Polysulfides are flexible, chemically resistant elastomers , with a linear polymer structure consisting of aliphatic carbon functional groups separated by one or more sulfur atoms. They are available as either one or two-part adhesives. One part adhesives cure slowly and are used primarily as sealants in the construction industry. Two-part adhesives are composed of polysulfide and epoxy resins. They are less brittle than epoxy adhesives and have greater impact resistance and higher elongation. Their tensile strength is lower, however, and they possess a disagreeable odor that is characteristic of polysulfides. Due to their exceptional fuel resistance, they are widely used in the aerospace industry[7]. Polysulfides cure by a cross-linking reaction using inorganic oxidizing agents and polyvalent metals. Joints have reasonable shear and peel strengths and excellent environmental durability[19][25].

Advantages/disadvantages: Advantages include excellent environmental resistance, high levels of resistance to organic liquids including fuels, good flexibility at low temperature (– 62˚C; –80˚F) and legible shrinkage during cure. Disadvantages include relatively low cohesive strength, unpleasant odor, creep under loading and very little strength above 121˚C (250˚F)[26].

Sealants based on polysulfide liquid polymers originally found wide acceptance for applications requiring a flexible, adhering, chemically resistant composition of matter. Since they were the first liquid polymers available that could be cured at room temperature, they were soon specified for a number of military applications. Their use as aircraft sealants for fuel tanks still remains as a major outlet. Other military applications included a quick hose repair compound, a sealant for bolted steel tanks for quick assembly on the battle front, electrical potting compounds, caulks for wooden flight decks which were designed as a stop gap in the early days of aircraft carriers, sealing and adhering methacrylate bubbles on aircraft, sealing cocoons in the mothball fleets, adhering aluminum strips on wings for reducing air turbulence during flight, and many others. Areas where the polysulfide sealants are still used include dental impression compounds, cast printing rolls, casting compounds for flexible molds, electrical potting, and miscellaneous adhesives. Poly sulfide sealants are excellent adhesives and adhere to a wide variety of surfaces; but price inevitably plays a major role in the final selection of material used for high-volume applications[17].

2.4.1 Applications of polysulfide Rubber

For sealing structural floor joints in various applications such as:

1-Parapet wall joints[19].

2-Joints in metal and concrete sea walls.

3-Joints in water retaining structures (including potable water when using gun grade).

4-Runway aprons and runways

5-Road and tile joints.

6-Structural Floor Joints.

7-Expansion and construction joints.

2.5 Chemistry of Polysulfide Rubber

2.5.1 Monomers of Polysulfide Rubber

The polysulfide polymers are prepared by condensation of organic polyhalides with inorganic polysulfides in aqueous suspension. The principal monomer is bis-Zchloroethyl formal. Ethylene dichloride is used in special polymers. Generally, most products contain some cross-link precursor, e.g. 1,2,3-trichloropropane. Bis-4chlorobutyl formal and bis-4-chlorobutyl ether are used in small amounts where improvement in low-temperature performance is required. Properties of important dihalides used in the preparation of polysulfide polymers are shown in Table 2.3. Sodium disulfide is primarily the alkali polysulfide used although some special polymers may contain structures, e.g monosulfide or tetra sulfide[27].

2.5.2 Polymerization Reactions and Processes

The polysulfide polymers are prepared as suspensions by condensation of the dihalide monomer with polysulfide in aqueous solution[17]. Bis-Z chloroethyl formal is the usually used monomer and 0.1-4.0% 1,2,3,4richloropropane is added as a cross-linking agent. In conducting polymerization, the standard practice is to feed the dihalide monomer into the aqueous polysulfide solution containing specific suspending and nucleating agents. A combination of analkyl naphthalene sulfonate with magnesium hydroxide sol prepared in situ is commonly used. The polymerization reaction that produces high molecular weight polymer is

$$nClC_2H_4OCH_2OC_2H_4Cl+nNa_2S_{2.25} \longrightarrow (C_2H_4OCH_2OC_2H_4S_{2.25})_n+2nNaCl$$

The sulfur is present as a mixture of disulfide and tri sulfide. Liquid polysulfide polymers which are commercially more important than the solid elastomers range from a molecular weight of 1000 to 8000. They are prepared from the high molecular weight polymer described earlier. High molecular weight polymer is split into segments which are simultaneously terminated by mercaptan groups:

$$RSSR+NaSH+NaHSO_3 \longrightarrow 2RSH+Na_2S_2O_3$$

Table 2.2: Properties of Dihalides.

Monomer	**Formula**	**BP (˚C)**	**η**
Bis-2-chloroethyl formal	$CH(OCH_2CH_2Cl)_2$	105-106/14	1.4557
1,2,3-Trichloropropane	CH_2Cl $CHCl$ CH_2Cl	156	1.4835
Ethylene dichloride	CH_2Cl CH_2Cl	83.6	1.4443
Bis-4-chlorobutyl ether	$O(CH_2CH_2CH_2CH_2Cl)_2$	125-128/12	1.4589
Bis-4-chlorobutyl formal	$CH_2O(CH_2CH_2CH_2CH_2Cl)_2$	154-156/3	1.4066

The concentration of the splitting salts regulates the average molecular weight of the liquid polysulfides. For example, Thiokol LP-2 has the following average formula:

$HS\ (-C_2H_4OCH_2OC_2H_4SS-)_{23}C_2H_4OCH_2OC_2H_4SH$

2.5.3 Copolymerization of polysulfide

Using of a mixed dihalide monomer feed in a conventional polymerization will produce random copolymers. A block copolymer cannot be prepared by step wise addition of the monomers. During copolymerization, interchange takes place, resulting in randomization. To prepare block copolymers it is best to prepare individual mercaptan terminated polymers, blend them in the desired proportion, and then cure the blended system by conventional techniques[27].

2.6 Theories of Adhesion

2.6.1 Mechanical Theory

According to this theory, adhesion occurs by the penetration of adhesives into pores, cavities, and other surface irregularities on the surface of the substrate. The adhesive displaces the trapped air at the interface. Therefore, it is concluded that an adhesive penetrating into the surface roughness of two adherends can bond them. A positive contribution to the adhesive bond strength results from the " mechanical interlocking " of the adhesive and the adherends. Adhesives frequently form stronger bonds to porous abraded surfaces than they do to smooth surfaces. However, this theory is not universally applicable, since good adhesion also takes place between smooth surfaces. Enhanced adhesion after abrading the surface of an adherend may be due to (1) mechanical interlocking, (2) formation of a clean surface, (3) formation of a highly reactive surface, and (4) an increase in contact surface area. It is believed that changes in physical and chemical properties of the adherend surface produce an increase in adhesive strength[28]. It can be debated whether mechanical interlocking is responsible for strong bonds or an increase in the adhesive contact surface enhances other mechanisms. More thorough wetting and more extensive chemical bonding are expected consequences of increased contact surface area.

2.6.2 Electrostatic (Electronic) Theory

This theory proposes that adhesion takes place due to electrostatic effects between the adhesive and the adherend[29-32]. An electron transfer is supposed to take place between the adhesive and the adherend as a result of unlike electronic band structures. Electrostatic forces in the form of an electrical double layer are thus formed at the adhesive adherend interface. These forces account for the resistance to separation. This theory gains support from the fact that electrical discharges have been noticed when an adhesive is peeled from a substrate[28].The electrostatic mechanism is a plausible explanation for polymer–metal adhesion bonds. The contribution of the electronic mechanism in nonmetallic systems to adhesion has been calculated and found to be small when compared with that of chemical bonding[33 and 34].

2.6.3 Diffusion Theory

This theory suggests that adhesion is developed through the interdiffusionof molecules in between the adhesive and the adherend. The diffusion theoryis primarily applicable when both the adhesive and the adherend arepolymers with relatively long-chain molecules capable of movement. Thenature of materials and bonding conditions will infl uence whether and towhat extent diffusion takes place. The diffuse interfacial (interphase) layertypically has a thickness in the range of 10 – 1,000 Å(1–

100 nm). Solventcementing or heat welding of thermoplastics is considered to be due to diffusion of molecules[28].

No stress concentration is present at the interface because no discontinuityexists in the physical properties. Cohesive energy density (CED, Eq. (1.1))can be used to interpret diffusion bonding, as defi ned by Eq. (1.2) . Bondstrength is maximized when solubility parameters are matched betweenthe adhesive and the adherend.

Eq. ˙(1) $$CED = \frac{E_{coh}}{V}$$ ………………………………….[19]

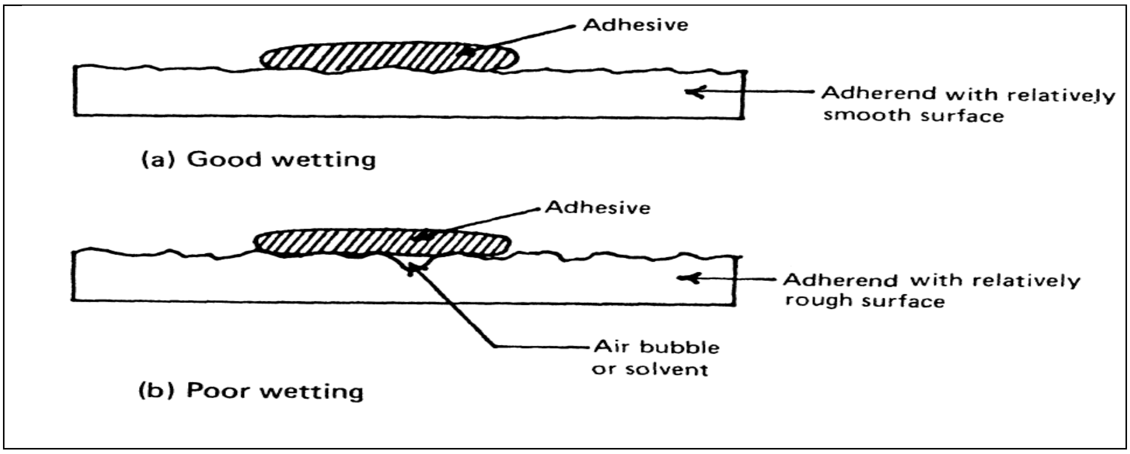
Eq. (2) $$\delta = \sqrt{\frac{E_{coh}}{V}}$$ ……………………………….[19]

2.6.4 Wetting Theory

This theory proposes that adhesion results from molecular contact betweentwo materials and the surface forces that develop. The first step in bondformation is to develop interfacial forces between the adhesive and thesubstrates. The process of establishing continuous contact between theadhesive and the adherend is called wetting . For an adhesive to wet a solidsurface, the adhesive should have a lower surface tension than the criticalsurface tension of the solid. This is precisely the reason for surface treatmentof plastics, which increases their surface energy and polarity.

Figure(2.4) illustrates complete and incomplete wetting of an adhesivespreading over a surface. Good wetting results when the adhesive fl owsinto the valleys and crevices on the substrate surface. Poor wetting resultswhen the adhesive bridges over the valley and results in a reduction of theactual contact area between the adhesive and the adherend, resulting in alower overall joint strength[28]. Incomplete wetting generates interfacialdefects, thereby reducing the adhesive bond strength. Complete wettingachieves the highest bond strength.

Most organic adhesives readily wet metal adherends. On the other hand,many solid organic substrates have surface tensions lower than those ofcommon adhesives. The criteria for good wetting requires the adhesives tohave a lower surface tension than the substrate, which explains, in part, whyorganic adhesives such as epoxies have excellent adhesion to metals butoffer weak adhesion on untreated polymeric substrates such aspolyethylene,polypropylene, and fluoroplastics [28].The surface energy of plastic substratescan be increased by various treatment techniques to allow wetting.



Figure(2.4): Examples of good and poor wetting by an adhesive spreading across a surface. Modified after Schneberger[28].

2.7 Mechanisms of adhesion

The mechanisms of adhesion are still not fullyunderstood and many theories are to be found inthe current literature. Often, the proponents ofeach theory ot'fer their hypothesis as a comprehensiveexplanation of all adhesion phenomenaand exclude all the alternative explanations. Muchof this confusion undoubtedly arises because thetest methods commonly employed to measure thestrengths of adhesive joints are not well suited totheoretical analysis. They introduce geometricalfactors and loading factors which are difficult toanalyse, and the measured joint strength includesindeterminate contributions from theologicalenergy losses in the adhesive and substrate. Thus,although the intrinsic adhesion forces acting acrossthe adhesive/substrate interface may affect jointstrength they are usually completely obscured byother contributions, and information concerningthe magnitude of such forces may only be indirectlyobtained. This inability to measure the interfacialinteractions has been the main obstacle to the developmentof a comprehensive theory of adhesion.The four main mechanisms of adhesion which havebeen proposed are[35]:

1. mechanical interlocking.

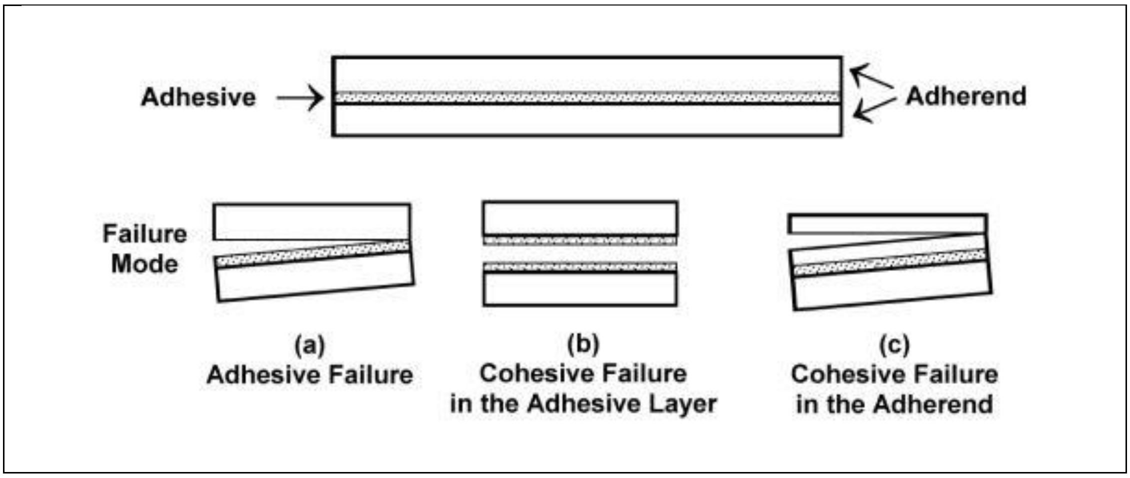
2. diffusion theory.

3.electronic theory.

4.adsorption theory.

2.6 Mechanisms of Bond Failure

Adhesive joints may fail adhesively or cohesively.Adhesive failure is an interfacial bond failurebetween the adhesive and the adherend. Cohesivefailure occurs when a fracture allows a layer ofadhesive to remain on both surfaces. When theadherend fails before the adhesive, it is known asa cohesive failure of the substrate. Various modes offailure are shown in Figure(2.5). Cohesive failurewithin the adhesive or one of the adherends is theideal type of failure because with this type of failurethe maximum strength of the materials in the jointhas been reached. In analyzing an adhesive joint thathas been tested to destruction, the mode of failure isoften expressed as a percentage cohesive or adhesivefailure, as shown in Figure(2.5).The ideal failure isa 100% cohesive failure in the adhesion layer.The failure mode should not be used as the onlycriterion for a useful joint[36].Some adhesiveeadherend combinations may fail adhesively, butexhibit greater strength than a similar joint bondedwith a weaker adhesive that fails cohesively. Theultimate strength of a joint is a more importantcriterion than the mode of joint failure. An analysisof failure mode, nevertheless, can be an extremelyuseful tool in determining whether the failure wasdue to a weak boundary layer or due to impropersurface preparation.The exact cause of premature adhesive failure isvery difficult to determine. If the adhesive does notwet the surface of the substrate completely, the bondstrength is certain to be less than maximal. Internalstresses occur in adhesive joints because of a naturaltendency of the adhesive to shrink during setting, andbecause of differences in physical properties ofadhesive and substrate. The coefficient of thermalexpansion of adhesive and adherend should be asclose as possible to minimize the stresses that maydevelop during thermal cycling or after cooling froman elevated temperature cure. Fillers are often used tomodify the thermal expansion characteristics ofadhesives and limit internal stresses. Another wayto accommodate these stresses is to use relativelyelastic adhesives.The types of stress acting on completed bonds,their orientation to the adhesive, and the rates atwhich they are applied are important factors indetermining the durability of the bond. Sustainedloads can cause premature failure in service, eventhough similar unloaded joints may exhibitadequate strength when tested after aging. Someadhesives break down rapidly under dead load,especially after exposure to heat or moisture. Mostadhesives have poor resistance to peel or cleavageloads. A number of adhesives are sensitive to therate at which the joint is stressed. Rigid, brittleadhesives sometimes have excellent tensile or shearstrength but have very poor impact strength.Operating environmental factors are capable ofdegrading an adhesive joint in various ways. Ifmore than one environmental factor (e.g., heat andmoisture) is acting on the sample, their combinedeffect can be expected to produce a synergisticresult of reducing adhesive strength. Wheneverpossible, candidate adhesive joints should be evaluatedunder simulated operating loads in the actualenvironment the joint is supposed to encounter[37].



Figure(2.5): Schematics of adhesive bond failure modes[37].

2.9 SurfaceTreatment for Adhesive

There are a number of reasons for surface treatment of materials. The main reasons for applying surface treatments prior to bonding are as follows[37]:

1-To remove or prevent the later formation ofa weak layer on the surface of the substrate.

2- To maximize the degree of molecular interaction between the adhesive or primer and the substrate surface.

3- To optimize the adhesion forces that develop across the interfaces to ensure sufficient initial joint strength and during the service life of the bond.

4- To create specific surface microstructure on the substrate.

Normally, optimum surface energy and structure is achieved by chemical surface treatment. The chemical composition and the morphology of the surface are changed, so that the surface energy of the substrate is maximized for adhesion. Chemical treatments also increase the chances that hydrogen, dipole, van der Waals ionic and/or covalent bonding can form at the substrate/adhesive interface. A quick and dirty test for adequate chemical treatment is to place a bead of water on the surface of the part and note if the water spreads. If so, the contact angle it forms with the surface is very small. There are special fluids called dyne liquids that bracket the numeric value of the surface energy of a material surface and fairly precisely[37].

2.9.1 Degreasing

Removal of all traces of oily contamination and grease from the substrate surfaces to be bonded is vital to the formation of strong adhesive bonds. Different methods, which should be carried out even though the contact surfaces may appear clean, are available for degreasing. A part may be submerged in trichloroethylene or perchloroethylene vapors even though the vapors are pure uncontaminated solvent. As the vapors condense on the part, contaminants are dissolved and drip off the part with the condensed solvent. When a vapor degreasing unit is not available, the joint surfaces can be wiped with a cloth soaked with trichloroethylene, followed by complete evaporation from the joint surfaces. These solvents are toxic in both liquid and vapor form, requiring the work environment to be well ventilated[37].

2.9.2 Abrasion

Abraded rough surfaces usually form stronger adhesive joints than do highly polished surfaces, primarily due to larger contact surfaces. A properly abraded surface should not contain any smooth or polished areas. Abrasion treatment should be followed by a second degreasing treatment to ensure the removal of loose particles. Grit-blasting removes surface deposits such as tarnish, rust, or mill scale from metal surfaces. If grit-blasting equipment is not available, or the metal is too thin to withstand blast treatment, clean the joint surfaces with a wire-brush, emery cloth, or sandpaper. Painted surfaces should be stripped down to substrate with a stripper prior to preparation, otherwise the adhesive joint will not be strong[37].

2.9.3 Chemical Treatment

Degreasing alone, or degreasing followed by abrasion and further degreasing, is sufficient for many adhesive bonds. To obtain maximum strength, reproducibility and resistance to deterioration a chemical or electrolytic pretreatment is required. Careful attention should be paid in the preparation of chemical solutions to assure correct proportioning of components required for formation of adequate bond strengths. Exposure time in the solution application is critical. If the application is too short, it does not sufficiently activate the surfaces. Overexposure to the solution builds up a layer of chemical reaction products that may interfere with the adhesion bond formation[37].

2.10 Mechanism of Adhesive Bonding

In adhesive bonding, attractive forces form between the adhesive and the adherends. The type of attractive force varies with the type of adhesive and adherends, but is generally a combination of some or all of the following forces:

•*Adsorptive*: These forces result from intimate interaction between particles at the joint surface. Attachment of such particles (including atoms or molecules) can be through weak, dipolar, or Vander Waals interactions or through chemical, usually covalent bonds.

•*Electrostatic*: These forces are due to ionic bonds between oppositely charged species or molecules.

•*Diffusive*: These forces result from molecular chain entanglements between the adherend and adhesive as they diffuse across the joint interface.

It is not always appreciated that attractive interfacial forces are very strong. Indeed, in many cases these forces are stronger than the cohesive forces of the adhesive or plastic adherend, and joint failure generally occurs in the adhesive or adherend, rather than at the joint interface. Additionally, interfacial bond strength is enhanced through the effect of larger scale micro and macro surface effects (through etching, roughening, abrasion, etc) resulting in mechanical interlocking and frictional forces[14][38].

2.11 Classification of Waste

Wastes are numerous in kind and defy easy definition. It is difficult to evolve a universal classification for 'waste'. However, roughly there are three basic classification methods, which are combined to form a waste classification system[39].They are:

- Chemical classification.
- Thermal classification.
- Physical classification.

The last one is directly related to thermal processing requirements and focuses on the information needed to select, design, and operate a thermal processing system[40].

2.12 Waste Concrete

The generation of solid residues of hydraulic concrete, also considered waste is turning into an environmental problem. The construction material primarily manufactured is Portland cement, but one of the main problems is its high manufacturing temperature which generates pollutants. The use of grinded aggregates that come from the demolition of hydraulic concrete is used to generate recycled hydraulic concrete, a material that could lessen costs, decrease pollution and cheapen construction. Nevertheless, the elaboration of recycled concrete faces the search for optimal designs in order to achieve the highest mechanical performance under static and dynamic requests[41].

Among many other fields, environmental preservation is an area of civil engineering that could be supported by the use of recycled concrete, which minimizes the discharge of solid residues that pollute the environment, reuses materials that are considered waste and that do not have a significant cost, and innovates material design to achieve the maximum mechanical performance under static and dynamic requests that allows for health improvement of those who use buildings constructed with these materials. Recycled concrete also preserves the environment as it prevents pollution from solid residues, decreases the emissions of CO_2 into the air that we breathe and prevents the unnecessary extractions from quarries of geological materials preserving landscape architecture and the endemic flora and fauna. Each profession is morally obligated and responsible to contribute to the best of its ability to the improvement and preservation of the environment. Recycling concrete is therefore an important research topic in order to prevent global warming. Its design, elaboration, durability, performance, economy and viability are researched. The employment of recycled construction materials dates back to the 1940's as Europe had excessive amounts of debris due to bombardments. This debris was used as quarries for building reconstruction with successful results. The countries most affected were the United Kingdom and Germany. The publications of that time, mainly British, German and Russian, informed of the use of debris for the construction of new civil work. Much of the debris comprised ceramic material(bricks, ceramic from sanitary services)[41].

2.13 Types of Cements

There are over ten different types of cements that are used in construction purposes, and they differ by their composition and are manufactured for different uses. these are rapid hardening cement (RHC), quick-setting cement (QSC), low heat cement (LHC), sulphate-resisting cement (SRC), blast furnace slag cement (BFSC), high alumina cement (HAC), white cement (WC), coloured cement (CC), Portland cement (PC),pozzolanic cement (PzC), air-entraining cement (AEC),and hydrophobic cement (HpC)[9].

2.14 Literature Review

In 2015, VikasSrivastava ,et al. replaced of regular fine aggregate by demolition waste fine aggregate up to 20% is possible without much compromising the strength and workability. Replacement of regular coarse aggregate by demolition waste coarse aggregate up to 30% is possible, without much compromising the strength and workability[42].

In 2016,P.lukowskl. Introduced of polymers into the cement composites improved same of the properties of concretes and mortars. Therefore, the polymer-cement composites are successfully used in construction. The model of microstructure formation in cement composites modified with thermoplastic polymer (pre-mix modifiers) has already been developed and successfully implemented. However, the formation of microstructure in the case of epoxy-cement composites (containing post-mix modifier) demonstrates same peculiarities which should be taken into account when modelling the process[43].

In 2016,Alessandra F. Baldisseraa,et al. This study investigates the chemical degradation of composites based on an epoxy resin blend (with and without a hardener) and cement paste when exposed to CO_2, aiming to provide a new polymer-modified cement with better chemical resistance. Results showed that the S1(composite without hardener) composites did not have satisfactory performance under common conditions encountered in deep CO_2 disposal. The epoxy resin could leach out of the cement matrix in the absence of a hardener, undermining the chemical resistance of cementitious materials. However, in the presence of a hardener, the S2(composite with hardener) composites have shown an improvement on the resistance against CO_2 acidic attack compared to unmodified cement paste. The optimal epoxy resin content in the S2 composites was found to be up to 30% in order to reduce the cement degradation[44].

In 2017,Ibtihal A. Mahmood, et al. In this research, binary blends have been prepared from epoxy resin (EP) and different weight percentages of polysulfide rubber (PSR) (0%, 2.5%, 5%, 7.5 and 10%), and then compression, impact, and hardness tests were evaluated. The experimental results showed that the addition of polysulfide rubber in the epoxy resin decreased the compressive strength, Young's modulus, and hardness, while increased the impact resistance. It was found that the weight percentage 5% of polysulfide was the best percentage, which gives the best mechanical properties for the blend matrix. The advantage of this blend matrix is that, it mediated between the brittle properties of epoxy and the flexible properties of a blend matrix with the highest percentage of PSR. Short fibers (Carbon & Glass) with different volume percentage (2.5%, 5%, 7.5%, and 10%), were used to reinforce the best blend matrix obtained separately and randomly, and then the same mechanical tests conducted on these composites. The experimental results showed that the addition of fibers increased the compressive strength, Young's modulus, impact resistance and hardness. It was also observed that the composites materials reinforced with carbon fibers had significantly higher mechanical properties values than the composites materials reinforced with glass fibers[45].

In 2018,A A Naser , et al. This work thus aims to investigate the effects of reinforcing epoxy by using waste material (glass or porcelain) to prepare particulate composites as a form of coating material. The final results showed a general improvement in the adhesion properties of composites when adding both glass and porcelain; nevertheless, porcelain results were much better than those seen with glass as filler with epoxy. This result achieved the main objective of the current work in terms of developing a better low-cost coating material for concrete substrates to be used for construction application[5].

In 2018,Joseph Jean Assaad .attempted to develop specialty cement possessing improved tensile and bond properties resulting from inter-grinding clinker with elastomer polymeric latexes such as styrene-butadiene rubber (SBR) or polyvinyl acetate (PVA). This type of ready-to-use cement could be of particular interesting adhesive applications requiring enhanced bond to substrates (i.e., tile adhesives, patching mortars, water proofing slurries) as well as repair and injection works necessitating improved durability and adhesion to embedded steel bars. Test results demonstrated that latexes remained efficient after grinding to altercement properties, including higher workability and improved flexural and pull-off bond strengths[46].

Chapter Three

Experimental Part

3.1 General Introduction

This chapter includes:

1- Listing the used materials (Polysulfide Rubber, Cement and Waste Concrete) and mention their properties.

2- Describe the procedures used to prepare the substrates.

3- The experimental procedure, which followed to prepare the sheet.

4- Listing the required tests and the devices such as FTIR, Tensile, Hardness, Pull off adhesion and SEM.

3.2 The Used Materials

3.2.1 Polysulfide Resin

Polysulfide adhesive was purchased from the company DCP, Hilla, Iraq with the properties list in Table(3.1).Flexseal PS660 is a two part polysulfide sealant which when mixed, cures to form a flexible rubber seal. It has good adhesion to concrete, stone, metals and many other common building substrates.

Table 3.1: properties of the used polysulfide Flexseal PS660[47].

Property	**Data**
Color	Grey
Solids	100%, passess the lead requirements for the VOC content
Working life:	(1:30 - 3:30) hr @ 20^{o}C (40 – 60) min @ 35^{o}C
Application temperature	(5 – 50)°C
Setting time:	(36-48)hr @ 15^{o}C (15-20)hr @ 25^{o}C (10-15)hr @ 35^{o}C

Service temperature:	(-40 – 90)°C
Cure rate:	7 days @ 25°C in a typical10 mm x 10 mm joint. at colder temperatures the cure rate will be extended
UV resistance:	Good
Biological resistance:	Resist microbiological active situations
Service life:	20 years (when used in trafficked areas or other special environments the life may be reduced
Flammability:	Does not support combustion
Butt joints (movement in tension and compression)	25%
Lap joints (movement in shear)	50%
VOC:	< 10 g/ltr

3.2.2 Portland Cement

There are over ten different types of cements that are used in construction purposes, and they differ by their composition and are manufactured for different uses. Portland cement was purchased from the company Mass, Hilla, Iraq with Content in Table (3.2).

Table 3.2: properties of the used cement.

Constituent	Ordinary Portland cement% by Weight
CaO	64.64
SiO_2	21.28
Al_2O_3	5.60
Fe_2O_3	3.36
MgO	2.06
SO_3	2.14
N_2O	0.05

3.2.3 Waste Concrete

Waste concrete usually consists of sand and gravel, and particle size it has 75µm or less can reduce costs, reduce pollution and reduce construction cost.

3.3 Samples Preparation

3.3.1 Preparation Pure Polysulfide Rubber Resin (PSR)

The polysulfide resin was mixed with hardener (with ratio 95: 5) using mechanical stirrer for 15 minutes. Then the mixture was poured into the glass mold ,which was previously prepared, then left it for 7 day at room temperature to complete curing. Figure (3.1) explains the steps to prepare samples of pure polysulfide Rubber.

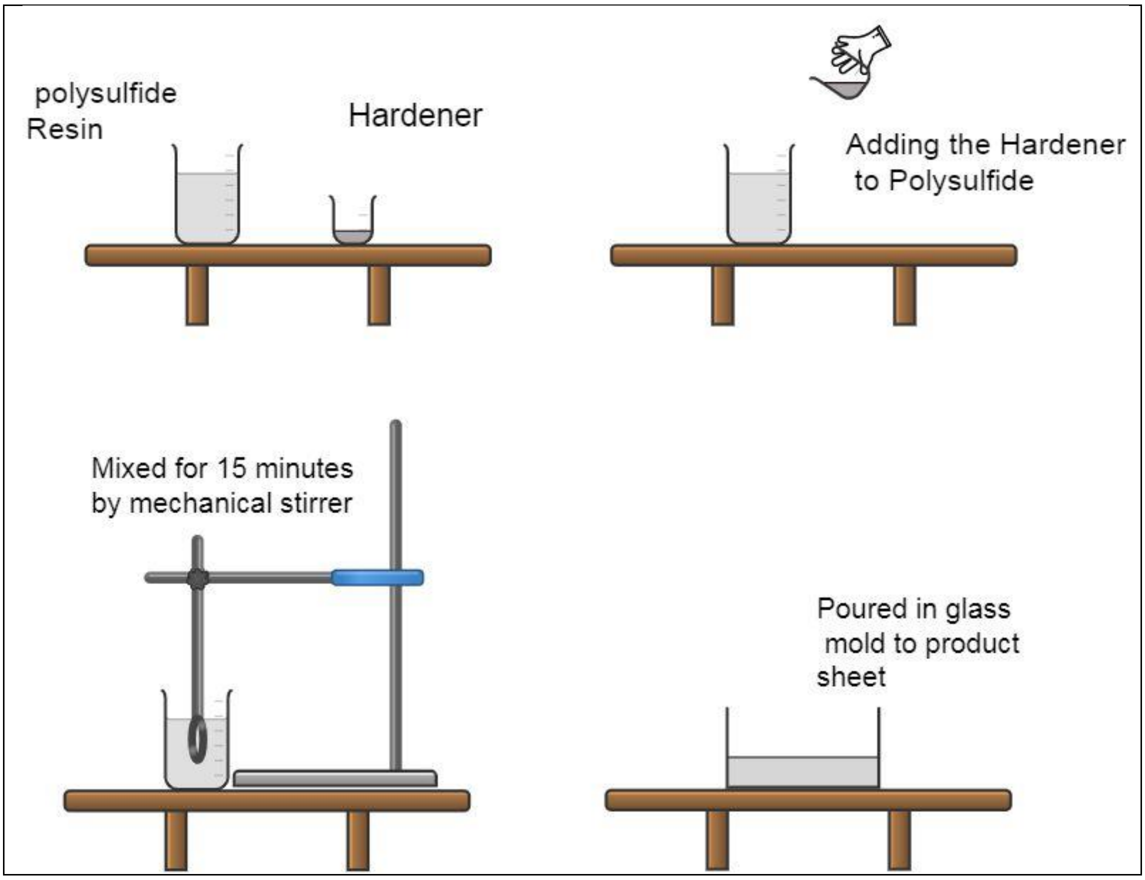


Figure (3.1):Preparation steps of pure polysulfide Rubber samples.

3.3.2 Preparation of Waste Concrete Particles

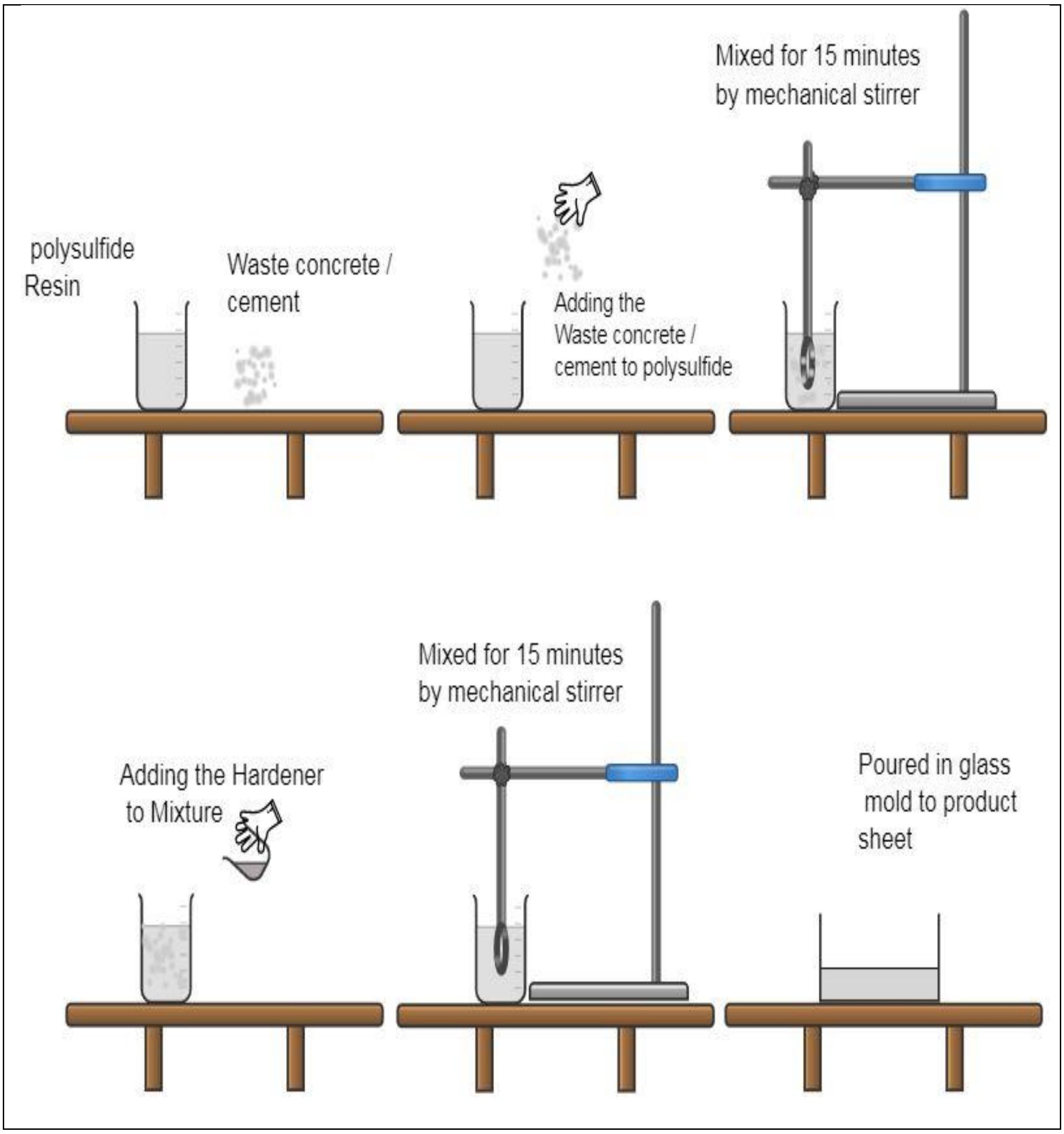
It was taken a ready made concrete cube and passed it in several stages, first cleaning it with water, then drying it with oven 80^{o}C and RT and grinding it first with a mortar until it became a fine powder. Then, the waste concrete was put it in the device (sieving) which was used to separate the large particles sizes from the small ones, where it was used at this stage the smallest sieving (0.075mm) to be inserted as particle of polysulfide as shown in figure (3.2).

Figure(3.2): Preparation of waste concrete.

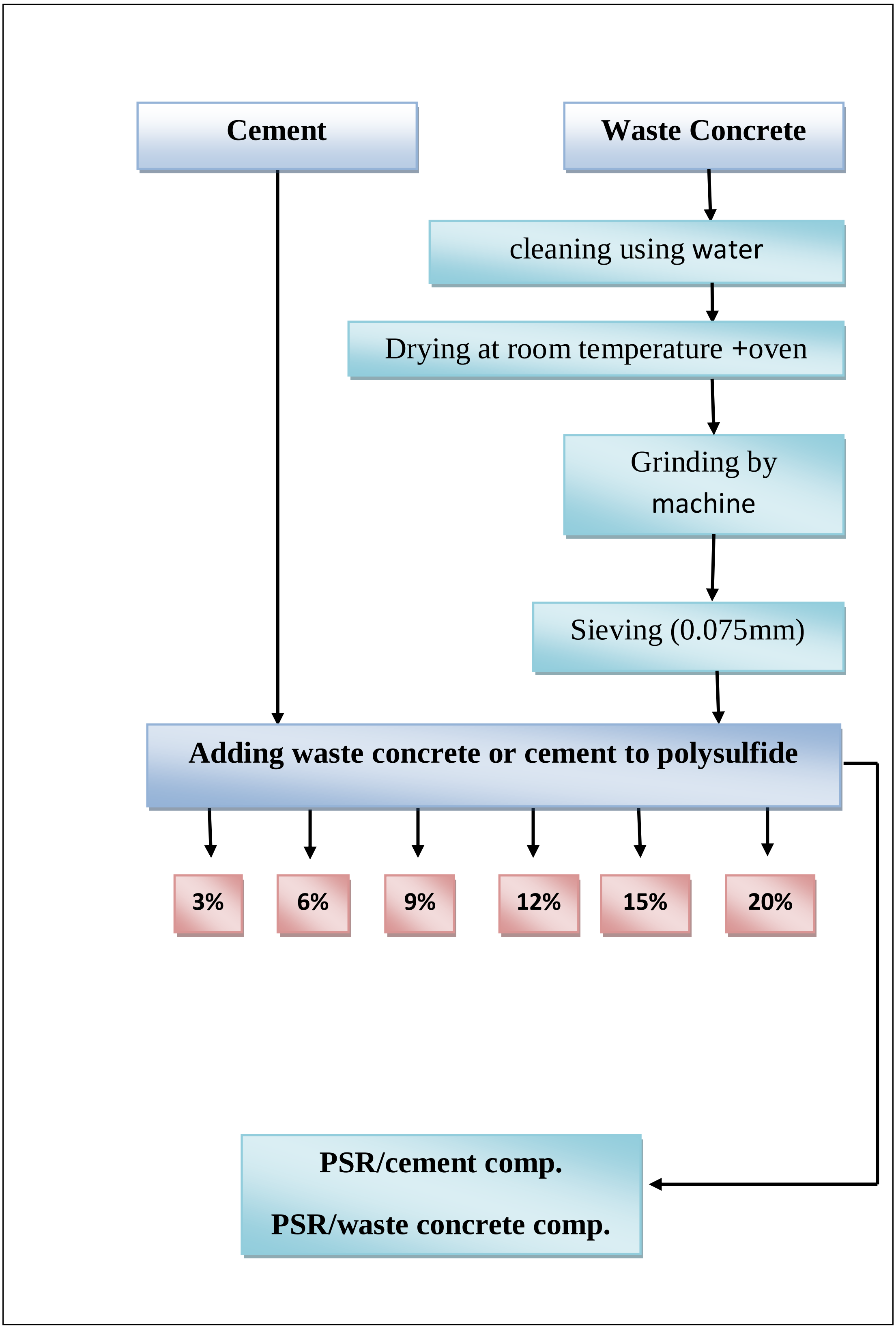
3.3.3 Preparation of Composite Samples

- Polysulfide Rubber / Cement Composites(PSR/C).
- Polysulfide Rubber / Waste Concrete Composite(PSR/W).

Composites were prepared using different parentages of particles cement or waste concrete (0, 3 , 6 , 9 , 12, 15 and 20wt %.) ,then the cement or waste concrete added into resin then using mechanical mixing for 10 min in order to get good mixing, then the hardener (with ratio 95:5) was added and was mixed for 15 minutes using a mechanical stirrer, and then the mixture was poured into the glass mold, and left it for 24 hours at room temperature. Figure (3.3) describes the procedures used to prepare the samples.



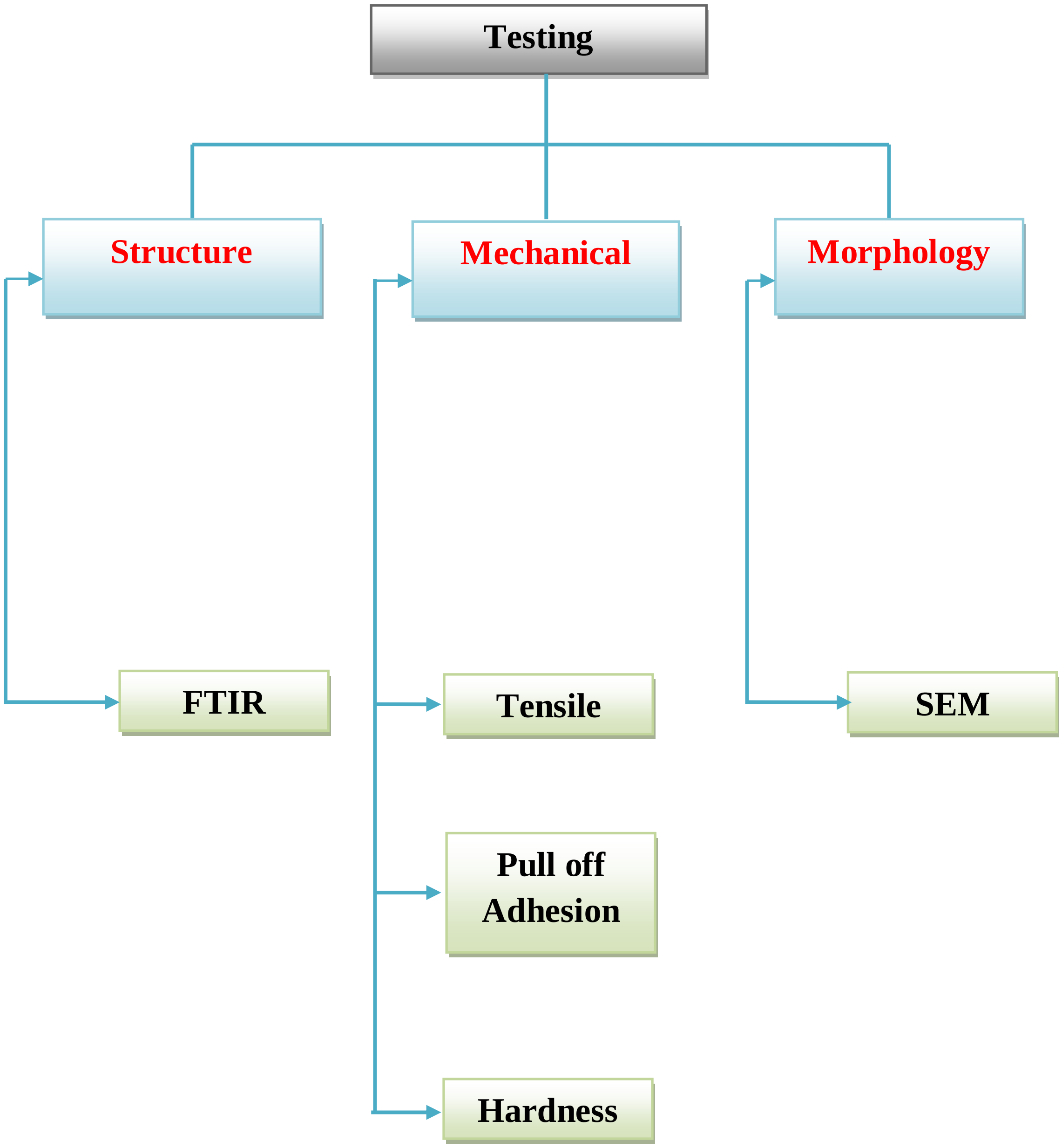
Figure(3.3): The procedure to prepare polysulfide rubber / cement or waste concrete composites.



Figure(3.4):Flow chart for plan work.

3.4 Characterization Tests

The preparation sheet film tested by using different techniques as shown in Figure (3.5).



Figure(3.5): Flow chart of testing.

3.5 Fourier Transform Infrared Spectrometer (FTIR) Test

Fourier transforms infrared technique used to characterize the prepared samples using instrument type (IR Affinity-1) made in (Kyoto Japan) located in laboratory of Polymer and Petrochemicals Department/ Materials Engineering collage /University of Babylon as shown in figure (3.7). In order to measure a sample, calibrate the device using the KBr, and then prepare a powder of the sample to be examined, and mixed with KBr (mixing ratio 99% KBr). FTIR spectrum provides a diagram between the permeability or absorption and the number of waves that show the chemical composition of the material. This test carried out according to ASTM E1252[48] for pure PSR ,PSR /W and PSR/ C in order to know the bond between the polymer chains and polymer chains and fillers if is a chemical or physical bond.

Figure(3.6):FTIR analyses devise.

3.6 Mechanical Tests

3.6.1 Tensile Test

Samples were cut according to the ASTM D638[49] Standard specimen dimensions. The machine used for the testing of tensile properties (Pure PSR or PSR/W or PSR/C) was micro computer controlled electronic universal testing machine model (WDW-5E) China, located in the laboratory of Materials Engineering collage /University of

Babylon. The test was conducted at applied load (5KN) and speed value (100mm/min) for all the samples at room temperature. Tensile stress was applied until the failure of the sample and stress-strain curve was obtained as shown in figure (3.7).

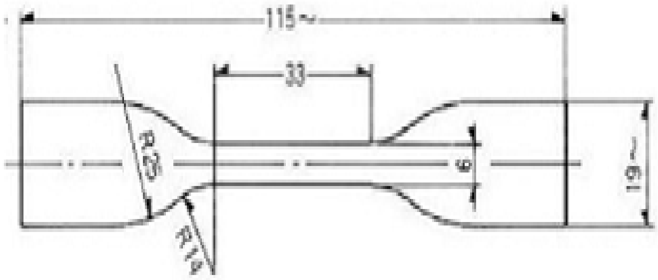
Figure(3.7): Universal testing machine.

3.6.1.1 Sample Cutting

After completing the process of casting samples in the glass molds and complete curing the samples, it was prepared the samples where the damper was used to cut of the samples and prepare them for mechanical tests such as shown in figure (3.8).

Figure(3.8): Samples after cutting.

Table 3.3: Sample tensile test dimension.

Property	**Sample**	**ASTM**
Tensile		D 638 IV

3.6.2 Pull Off Adhesion Strength Test

The general pull-off adhesion test is performed by scoring through the coating down to the surface of the concrete substrate at a diameter equal to the diameter of the loading fixture (dolly, stud), and securing the loading fixture normal (perpendicular) to the surface of the coating with an adhesive. The test was carried out under ASTM-D4541[50] Standard as shown in Figure (3.9).

Figure(3.9): Adhesion test samples.

After the adhesive was cured a testing apparatus was attached to the loading fixture and aligned to apply tension normal to the test surface. The force applied to the loading fixture was then uniformly increased and monitored until a plug of material is detached. When a plug of material was detached, the exposed surface represents the plane of limiting strength within the system. The nature of the failure is qualified in accordance with the percent of adhesive and cohesive failures, and the actual interfaces and layers involved. The pull-off adhesion strength is computed based on the maximum indicated load, the instrument calibration data, and the surface area stressed. Pull-off adhesion strength results obtained using different devices may be different because the results depend on instrumental parameters.

$$x = 4F/\pi d^2 \quad \text{..........} [50]$$

where:

X = pull-off adhesion strength achieved at failure in MPa(psi).

F = Maximum force applied to the test surface at failure in N(lbf).

d = diameter of the loading fixture in mm (in.).

2.6.2.1 Basic Steps to Perform Pull Off Test

1. Dolly & Coating Preparation: The dolly and the coating are cleaned and abraded.

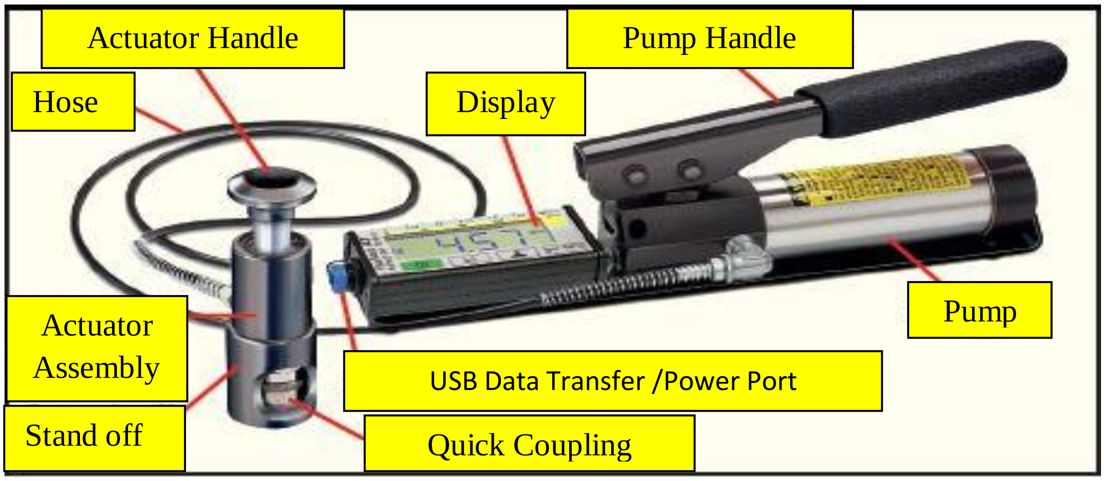
2. Glue & Dolly Application :The glue is prepared and applied to the dolly. The dolly is then adhered to the coated surface and the glue is allowed to cure.

3. Test Area Separation (optional): The test area of the coating is isolated from the are a surrounding the dolly by cutting or drilling.

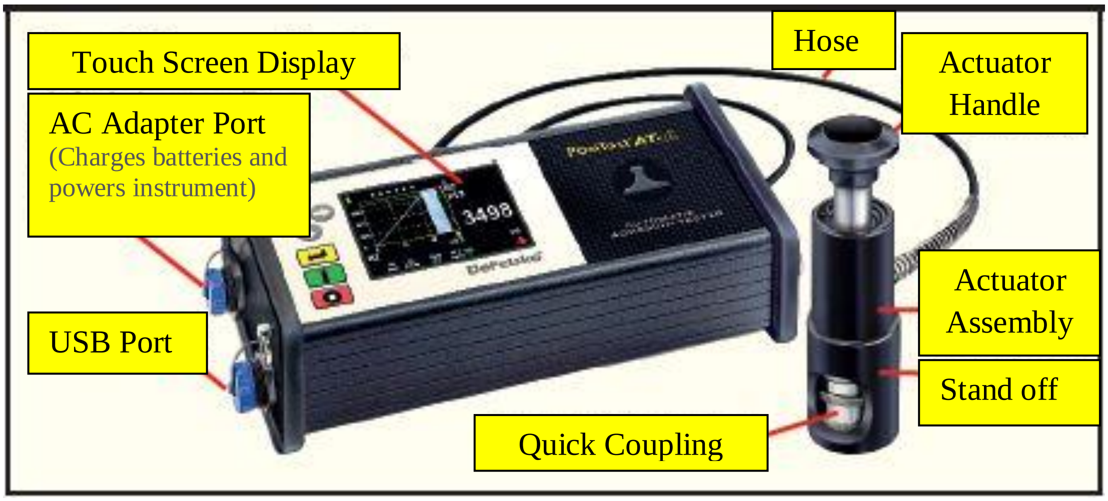
4. Pull-Off Test:

a) PosiTest AT-M (manual).

b) PosiTest AT-A (automatic).



Figure(3.10): PosiTest AT-M Manual.



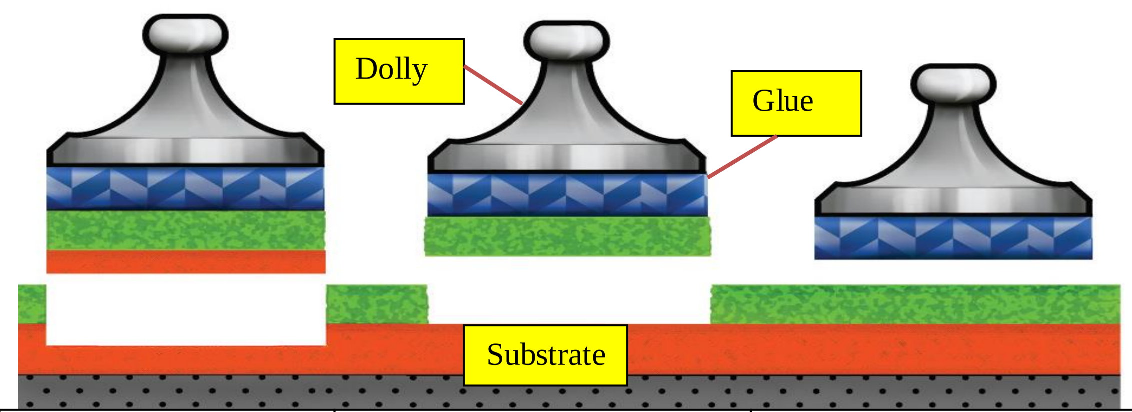
Figure(3.11): PosiTest AT-A Automatic.

5. Analysis of Test Results: Upon completion of the pull-off test, the dolly and coated surface should be examined. In addition to pull-off force, many National and International standards such as ASTM D4541 and ISO 4624require the nature of the fracture to be recorded:

1.Cohesive fracture:

2.Adhesive fracture:

3.Glue failure:



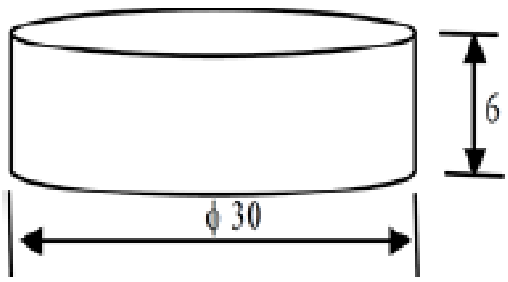
Cohesive fracture (Within a layer)	**Adhesive fracture** (between layers)	**Glue failure** (coating/glue)

Figure(3.12): Type of failure.

3.6.3 Hardness Test

The international hardness test is one of the measurements of the penetration of needle into the rubber specimen under specified conditions, the test was carried out under ASTM-D2240[51] Standard using durometer(Hardness shore A).Standardized hardness-measuring equipment using a sharp needle was applied directly on to the surface of (pure PSR or PSR/W or PSR/C) specimens to measure hardness. Data was averaged over six different positions as shown in Figure (3.13).

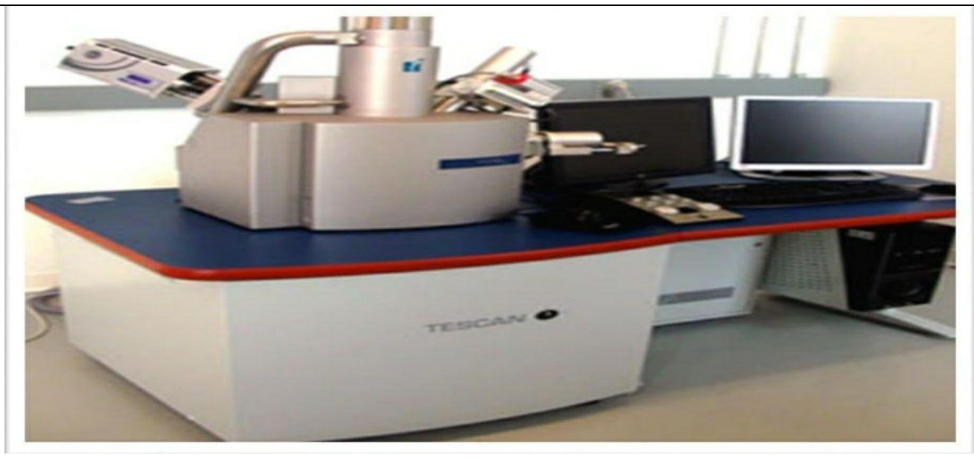
Table 3.4: Sample hardness test dimension.

Property	**Sample**	**ASTM**
Hardness (shore A)		D 2240

Figure(3.13): Hardness (shore A) test device.

3.7 Scanning Electron Microscopy (SEM) Test

Scanning electrons microscope is utilized powerful techniques that allow the observations and characterizations of the surfaces of both organic and inorganic material, providing important data concerning the morphologies of the sample. In this work was carried out to investigate the distribution of fillers in the PSR. The samples were tested using (SEM/ TESCAN/ VEGA II Series/ USA / Amir kaber University/ Iran) as shown in Figure (3.14).



Figure(3.14):SEM analyses machine.

Chapter Four

Results & Discussion

4.1 General Introduction

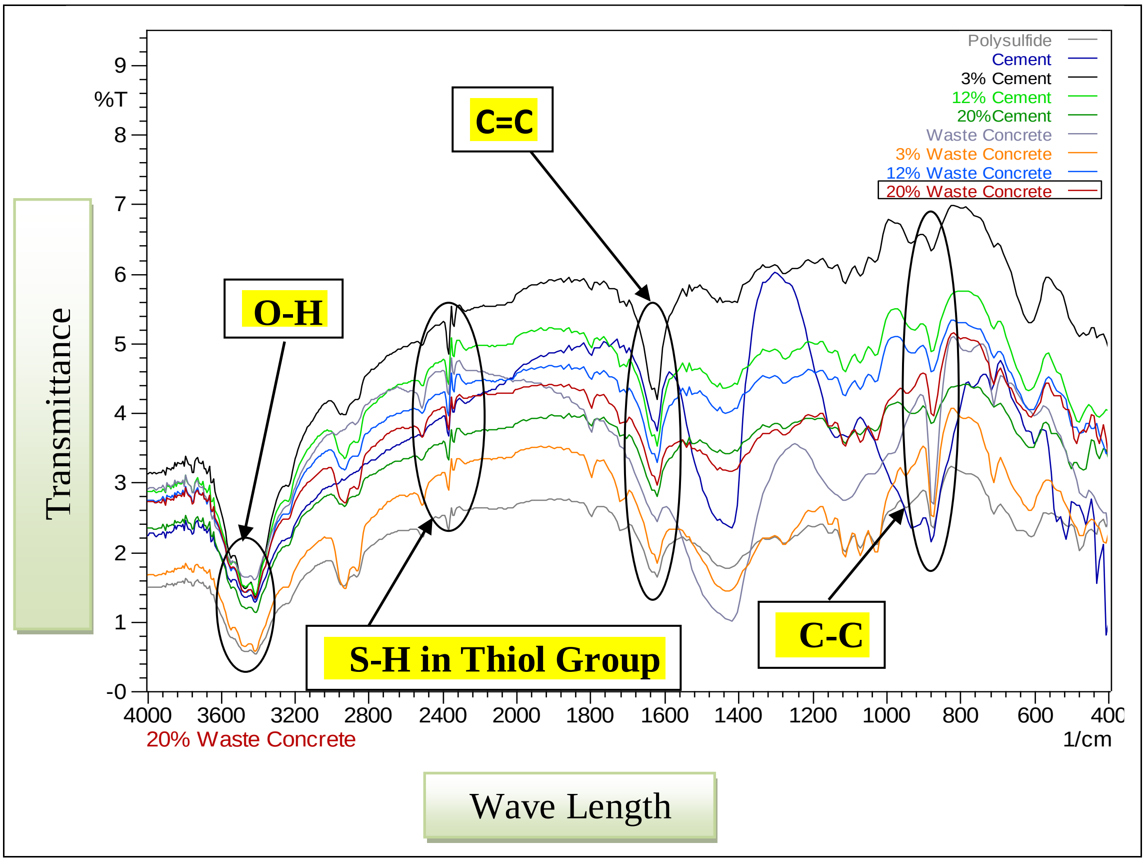
This chapter illustrates the results obtained through the results of structural, morphology and mechanical properties of Polysulfide Rubber (PSR) sheets with different concentrations of cement (C) and waste concrete (W) were studies in order to improve the adhesive of prepared sheets. This chapter includes the following :

1. Studying the structural properties by FTIR technique.
2. Studying the morphological properties by SEM technique.
3. Studying the mechanical properties of composite , by tensile, hardness (shore A) , pull off and adhesion techniques.

4.2 FTIR Results

The characteristic peaks of both the PSR polymer and the C and W were investigated by FTIR test. Band around 850 cm^{-1} represents the C-C bending mode. Band at 1700 cm^{-1} refers to the stretching vibration mode C=C for conjugated alkene structure within the PSR polymer. Because polysulfide Rubber is non polar. Weak band around 3500 cm^{-1} indicates the C-H bending mode in the aromatic compounds and strong band due to the O-H stretching mode. Band around 2550 cm^{-1} represents the S-H bending mode.

FTIR results also show that there is no chemical interaction between C or W and the polymer. There is no change because the interference of the particles is a physical interference, no bond was produced because concrete is a dead substance and cement is also, otherwise there would be a change in the shape of the polysulfide curve as show in Figure (4.1).



Figure(4.1):FTIR spectra of PSR and its composites with C and W.

4.3 Mechanical Results

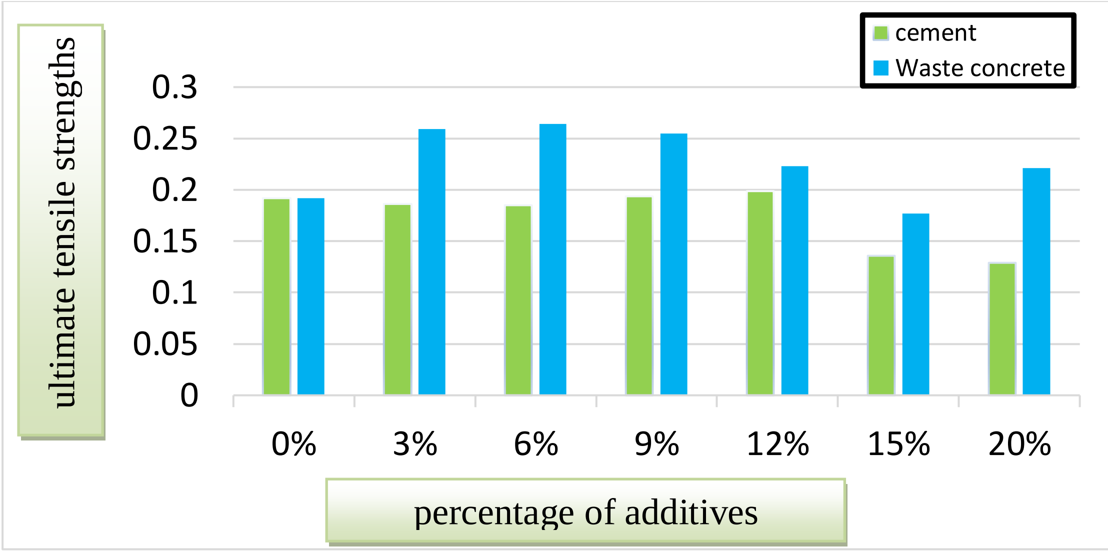
The mechanical results of composites (Pure PSR) (PSR/C), (PSR/W) include tensile , pull off adhesion and Hardness Tests.

4.3.1 Tensile Results

Figures (4.2) represents the relationship between the ultimate tensile strength and the percentage of additives (0, 3, 6, 9, 15 and 20 wt %) for each of the C and W of (a granular scale less than (75µm)).

Figure (4.2) shows the W amount effects on the tensile strength of PSR/W composites. The tensile strength of composite PSR/W increasing as W content increases except for (15%) in which there is a slight decrease within (0.177 MPa) after the tensile strength begins to rise with the increase in the percentage of addition, but it is higher or at the level of the tensile strength value of the pure polysulfide samples. The reason for the increase is due to the fact that W restricts the movement of the PSR chains due to the increase in the mechanical interlocking between the PSR chains and W particles , and also works to impede in the movement of chain significantly, thus increasing the cohesion. Agglomerates of W particles that results in the poor dispersion in PSR at high W content. That is mean the interaction between PSR and W is more favorable than PSR and C because to impede in the movement of chains significantly, thus increasing the cohesion.

Figure (4.2) shows the C amount effect on the tensile strength of PSR/C composites. As C was added to the composite, the tensile strength of the reinforced rubber with C is slight increase with the percentage of addition (12%)(0.199 MPa), but it is at the level of the tensile strength value of the polysulfide samples(non-reinforced) for the percentage lower than 12%. Then the tensile strength decreases at the ratio (15% and 20%) and the reason for the decrease is the occurrence of agglomeration of the particle, which leads to increase the space between the rubber chains and this leads to a decrease in the cohesion of the rubber and a reduce in the crosslink density of rubber and this appears significantly when the percentage of adding (15%, 20%). The restriction is not severe in the C because the size of the particle is of the type fine. In general ,PSR/W composites have better tensile strength than PSR/C composites because W particles give more severe restriction due to having bigger and rougher particles.

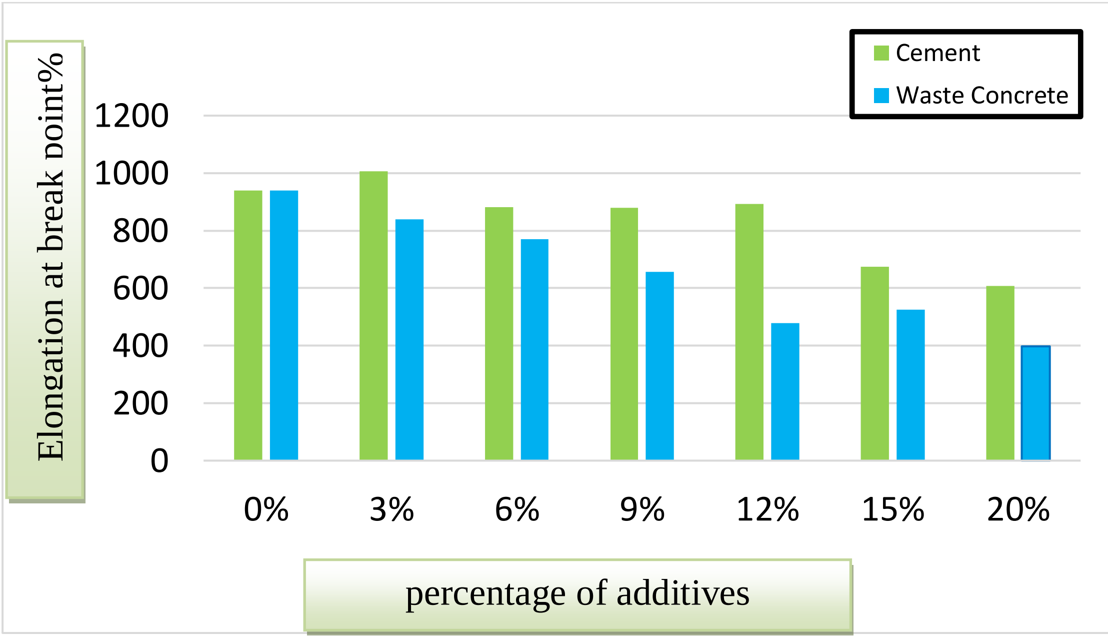


Figure(4.2): Comparison between the ultimate tensile strengths of polysulfide without and with different percentage of waste concrete and cement.

It could not be calculated the modulus of elasticity of PSR and their composites due to the behavior of rubber, as it has ultimate tensile strength very few limits (0.1 MPa - 0.2 MPa), meaning less than one and stress-strain curves appear as zikzik.

Figure (4.3)represents the relationship between the elongation and the percentage of addition (0, 3, 6, 9,12, 15 and 20 wt %.) for each of C and W.

The elongation of composites drops with the increasing in the W and C amount .The reduction in the elongation becomes more drastic in the PSR/W due to chains movement is more restricted by W resulting from their shape. In addition, the elongation of polysulfide is reduced from (940.08%) to (397.528%), meaning that it is reduced to (57%).The addition of C at a ratio of 20% to the polysulfide adhesive reduces the production cost. Moreover, the elongation of the polysulfide is decreased from (940.08%) to (606.684%), it reduces it to (%35). The reduction in the elongation of the PSR/C composite is lower than of the PSR/W composites due to C particles have finnier shape than W particles and restriction between PSR chains by C is less, and this means that the restriction is due to the morphology or the outer shape of the particle ,this causes the movement of the rubber chains to be less restricted, as compared to W particles that caused a significant restriction of the movement of the rubber chains leading to lower elongation. As shown in figure (4.8) the W particles is rough while in figure (4.11) the C particle is fine.



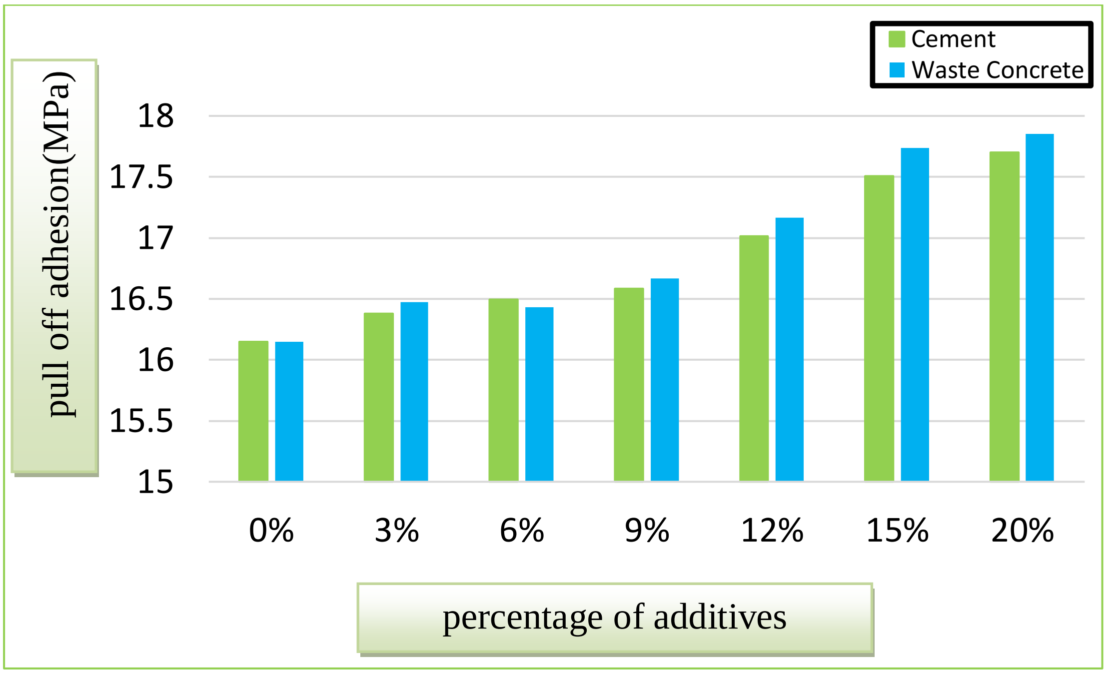
Figure(4.3): Comparison between the elongation at break point of polysulfide without and with different percentage of waste concrete and cement.

4.3.2 Pull-Off Adhesion Results

Figure (4.4) represents the relationship between the pull off adhesion strength and the percentage of addition (0, 3, 6 , 9 ,12 ,15 and 20 wt %) of C and W.

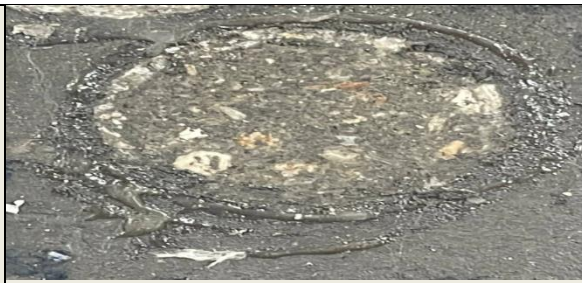
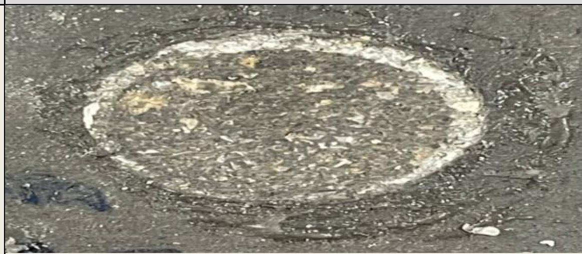
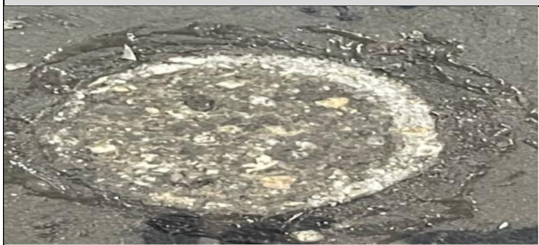
The addition of W or C particles at all percentage between 3% to 20% shows greater adhesion strength than pure PSR.

This is due to high percentages of C and W that leading to PSR liquid more rough when it wet the surface of substrate resulting in mechanical inter locking between W and C and substrate surface. As it be seen in figure(4.4).



Figure(4.4):Comparison between the pull off adhesion of polysulfide without and with different percentage of waste concrete and cement.

All pull- off adhesion test samples show adhesion failure and and as shown in figure (4.5).This is due to the cohesion force of the PSR and composites being greater than the adhesion force between the concrete and adhesive.

	
Pure polysulfide	Pure polysulfide
	
3% C	3% W
	
6% C	6% W
	
9% C	9% W
	
12% C	12% W

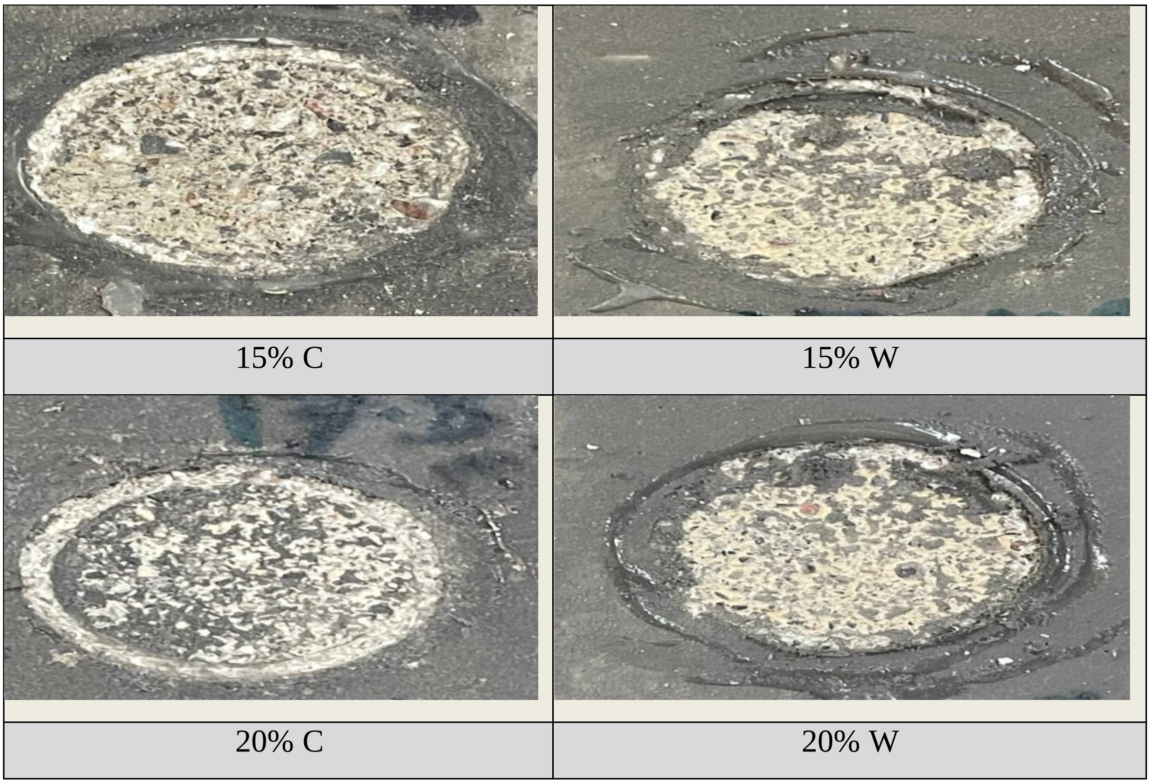


Figure (4.5): Image of adhesive failure shown on the concrete substrate.

4.3.3 Hardness Results

Figure (4.6) represents the relationship between the hardness (shore A) and the percentage of addition (0, 3, 6, 9, 12,15 and 20 wt %) for each of C and W.

In Figure (4.6) The values of reinforced PSR are either equal to or higher than the value of unreinforced polysulfide. The reason for this is the diffusion of the filler inside the material. It is noticed that the fluctuations in the results due to the possibility of agglomeration within the material, which leads to giving high hardness values in some areas. The hardness gives an indication that the filler impedes the penetration of the needle in the device of hardness.

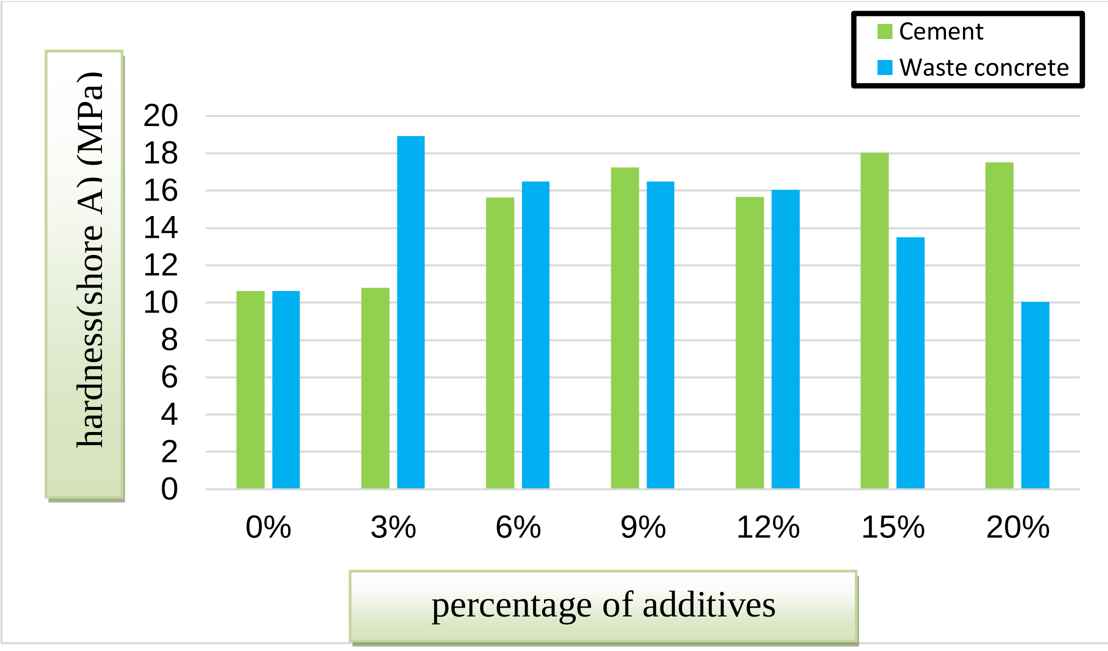
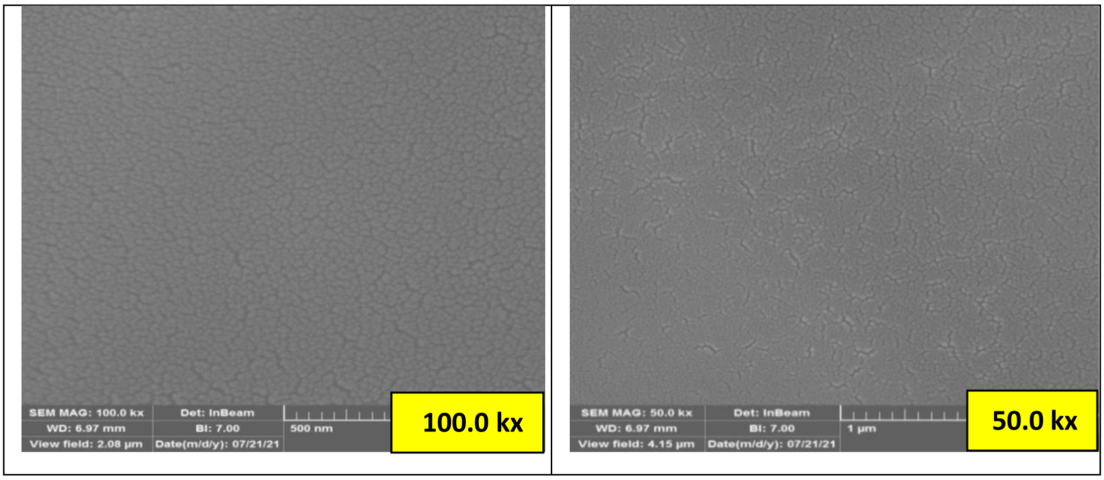


Figure (4.6): Comparison between the hardness(shore A) of polysulfide without and with different percentage of waste concrete and cement.

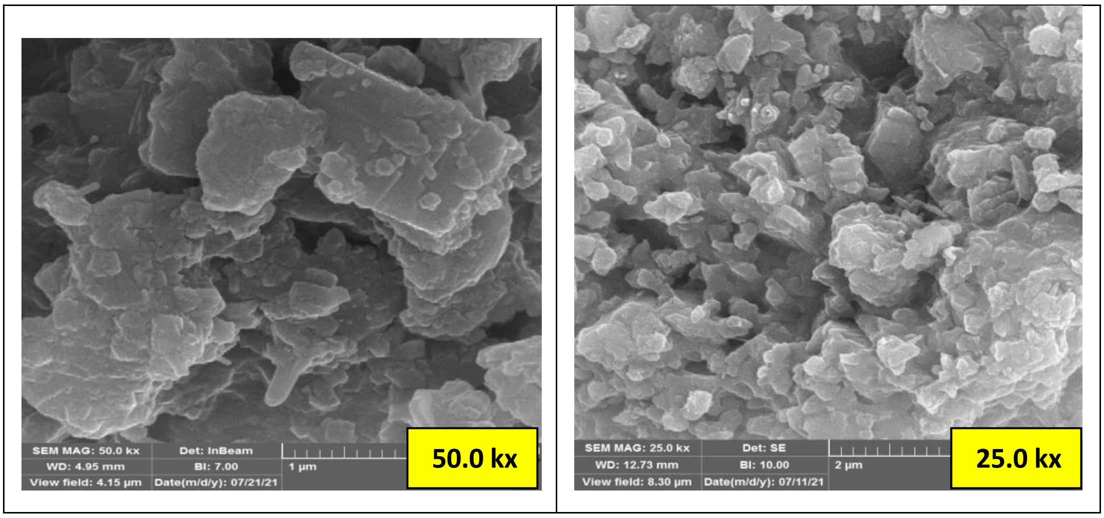
4.4 Scanning Electron Microscopy (SEM) Results

Figures (4.7 - 4.13) show the Scanning electron scope(SEM) images of PSR, W, C, PSR/W composite and PSR/C composite. Scanning electron micrographs (SEM) images of pure polysulfide , W and PSR/W composites are shown in Figure (4.8) to (4.10).SEM images show irregular and rough. SEM images of composites with W show that PSR cover W and an interconnected rough surface is produced.

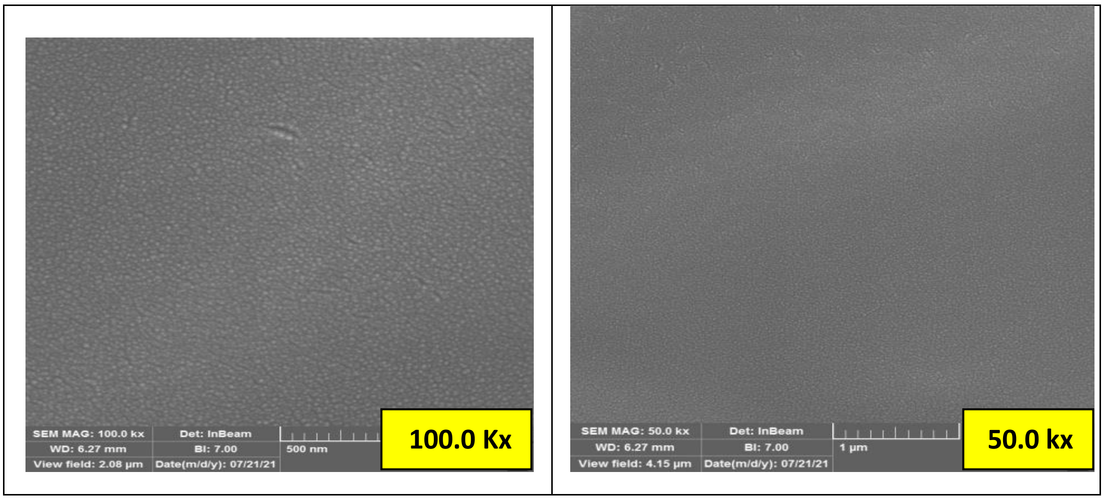
This means a good adhesion occur of polysulfide , this is due to W increase the adhesion through wetting and reduces the surface tension between PSR and W this agreed with mechanical properties. It can be seen that the shape of W particles is coarser and larger than particles C.



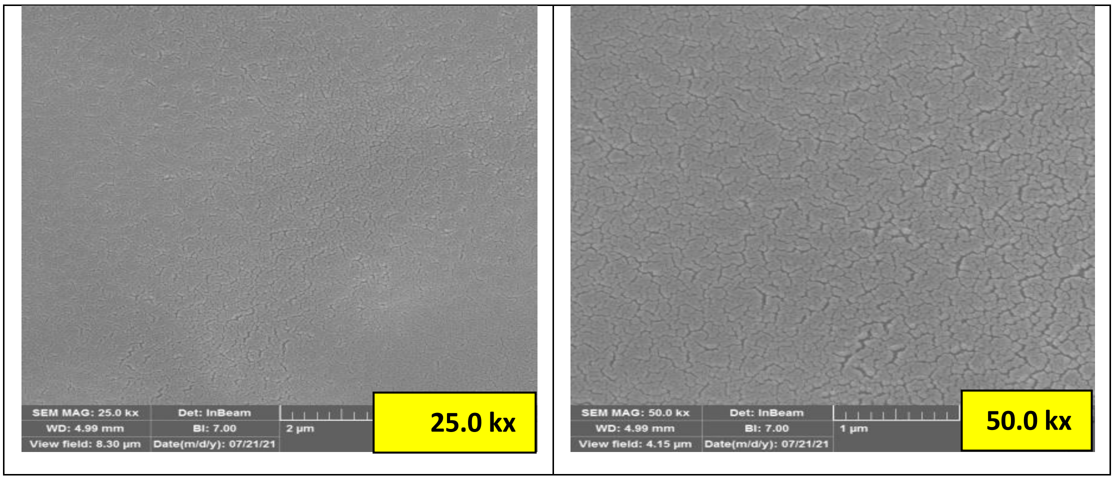
Figure(4.7): SEM images of pure PSR sample.



Figure(4.8): SEM images of W particles.

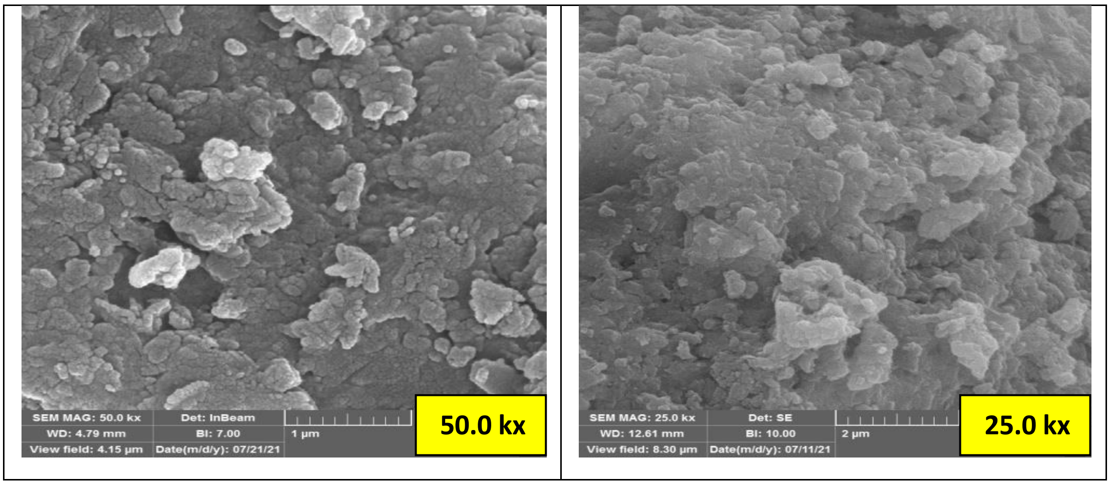


Figure(4.9): SEM images of 9% PSR/W.

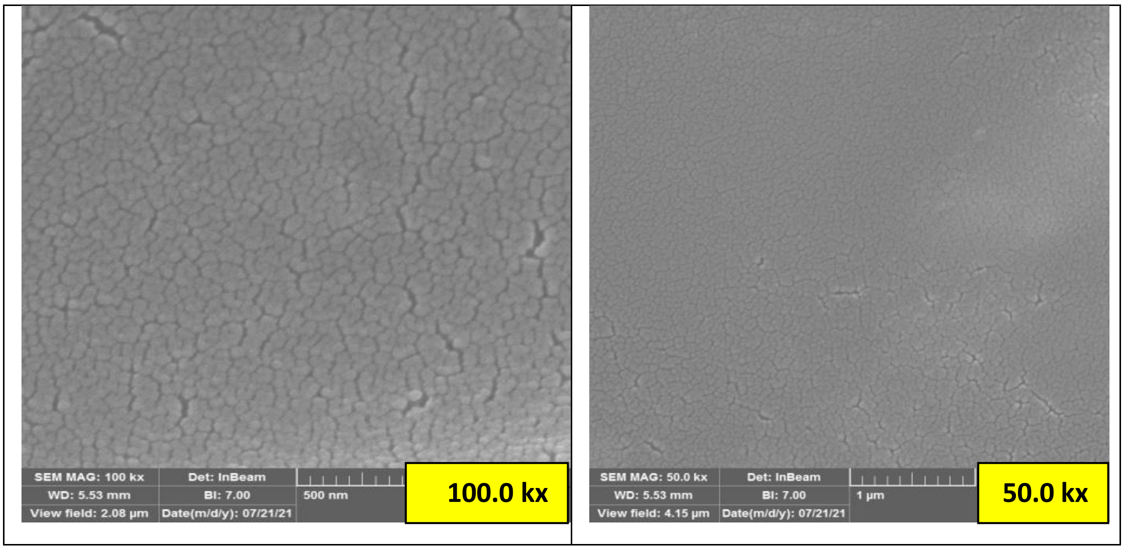


Figure(4.10): SEM images of 20% PSR/W.

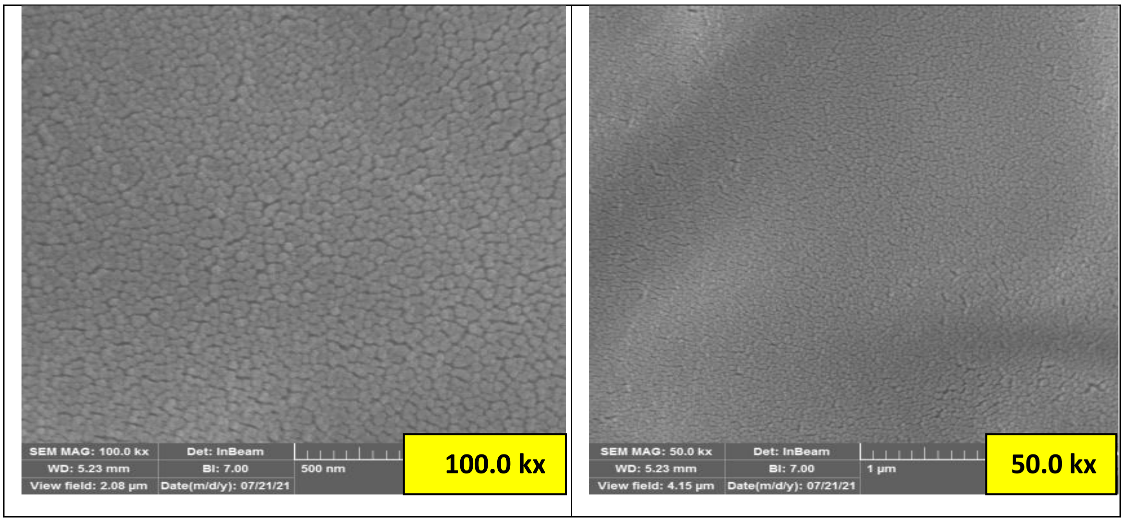
C particles have finnier size than W particles and restriction between PSR chains by C is less and this means that the restriction is due to the morphology or the outer shape of the particle, this causes the movement of the rubber chains to be less restricted as compared to W particles that caused a significant restriction of the movement of the rubber chains shown in Figure (4.11) to (4.13) images show fine size C partical.



Figure(4.11): SEM images of C particles.



Figure(4.12): SEM images of 9% PSR/C.



Figure(4.13): SEM images of 20% PSR/C.

Chapter Five

Conclusion& Recommendations

5.1 Conclusion

From the obtained results and their discussion in chapter four, the following conclusions can be drawn:

1) There is physical interaction between the polysulfide and cement or waste for FTIR test,.
2) Improve ultimate tensile strength increases for polysulfide compound with the increase of the waste concrete or cement.
3) The elongation of the composite materials decreases with the increase in the amount of addition of cement or waste concrete.
4) Pull off adhesion strength , shows improvement when adding the cement or waste concrete.
5) For hardness (shore A) Test, displays that the values of reinforced polysulfide are higher than that of pure polysulfide.
6) SEM , Images show an agglomeration in the waste concrete particles and their surfaces are a rough and irregular surface which is reflected in the mechanical properties. It can be seen that the shape of waste concrete particles is coarser and larger than cement. Cement particles have a finer shape than waste concrete particles.

5.2 Recommendations:

1) Increase the percentage of addition up to 40%.
2) Using another waste material like glass waste or porcelain waste.
3) Using epoxy for the same filler.
4) environmental effects such as UV and temperature.

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Appendix A

Table Appendix A.1: Analytical experimental avarage values obtained from the ultimatetensile strength PSR,PSR/C and PSR/W samples for 3 , 6, 9, 12, 15 and 20% at room temperature .

Addition Percent %	(Ultimate tensile strength)(N/mm^2)	
	Portland Cement	Waste concrete
0%	0.192	0.192
3%	0.186	0.259
6%	0.185	0.264
9%	0.194	0.255
12%	0.199	0.223
15%	0.136	0.177
20%	0.129	0.221

Table Appendix A.2: Analytical experimental avarage values obtained from the Elongation at break point % PSR, PSR/C and PSR/W samples for 3, 6, 9, 12, 15 and 20% at room temperature .

Addition Percent %	Elongation at break point %	
	Portland Cement	Waste concrete
0%	940.08	940.08
3%	1007.200	839.946
6%	882.311	770.847
9%	878.708	657.022
12%	892.857	478.844
15%	674.822	524.856
20%	606.684	397.528

Table Appendix A.3: Analytical experimental avarage values obtained from the adhesion tensile strength PSR,PSR/C and PSR/W samples for 3, 6, 9, 12, 15 and 20% at room temperature .

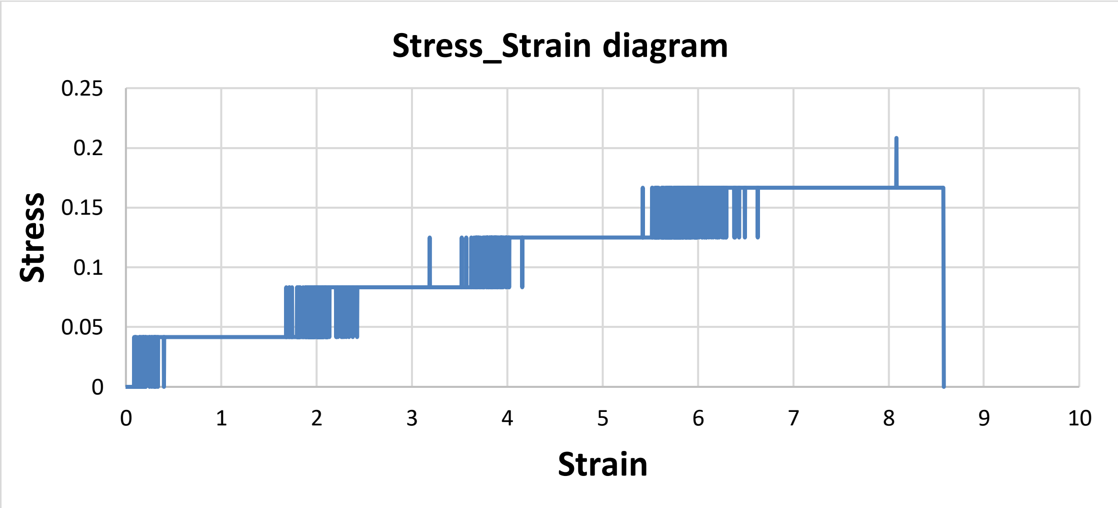
Addition Percent %	adhesion tensile strength(N/mm^2)	
	Portland Cement	Waste concrete
0%	16.15	16.15
3%	16.37	16.47
6%	16.49	16.43
9%	16.58	16.66
12%	17.01	17.16
15%	17.50	17.73
20%	17.69	17.85

Table Appendix A.4: Analytical experimental avarage values obtained from the hardness test (Shore A) PSR, PSR/C and PSR/W samples for 3, 6, 9, 12, 15 and 20% at room temperature.

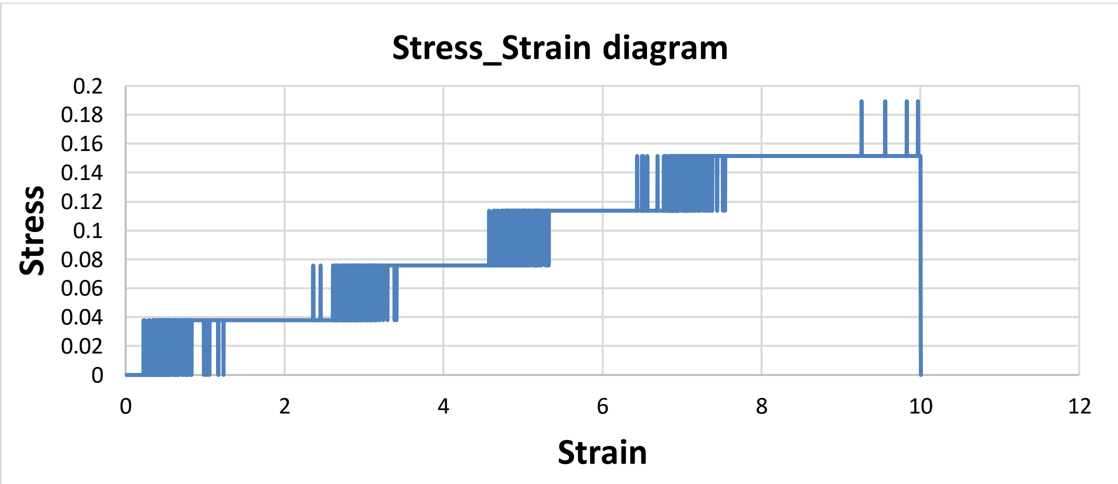
Addition Percent %	Hardness (Shore A)	
	Portland Cement	Waste concrete
0%	10.61	10.61
3%	10.78	18.91
6%	15.64	16.5
9%	17.25	16.5
12%	15.65	16.05
15%	18.02	13.5
20%	17.53	10.03

Appendix B

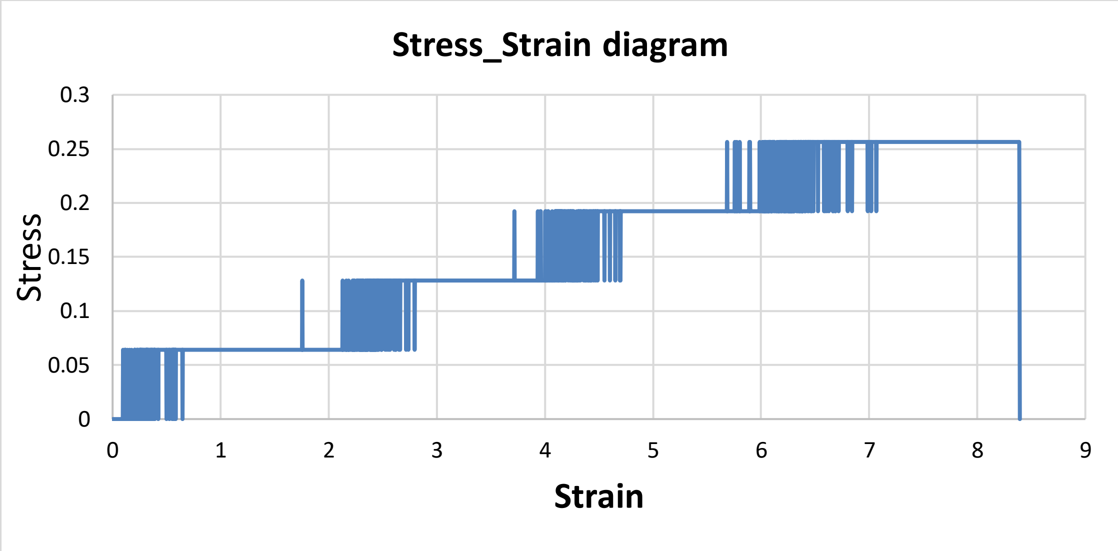
Table Appendix B.1: Stress-Strain curves obtained from the tensile test PSR, PSR/C and PSR/W samples for 3, 6, 9, 12, 15 and 20% at room temperature.



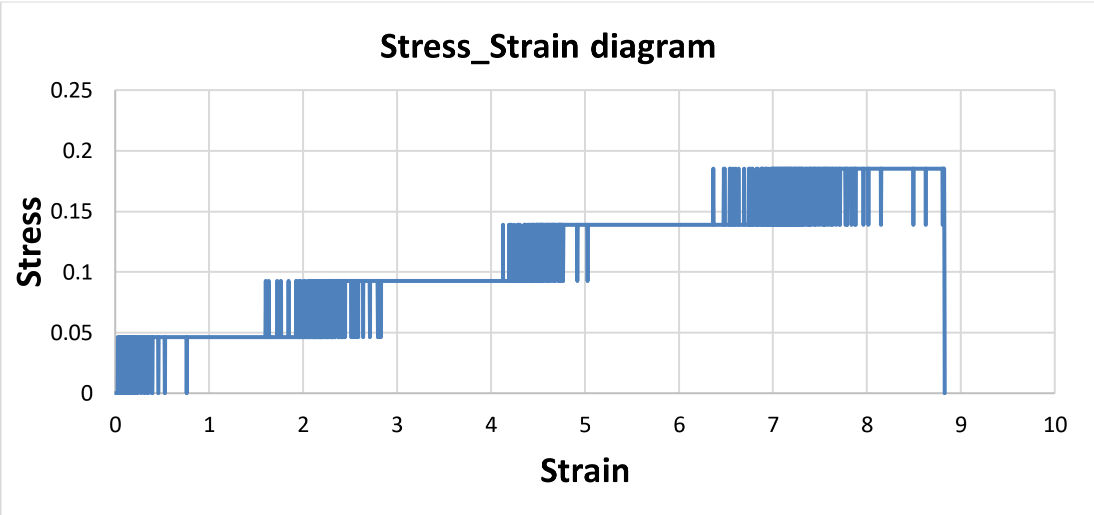
Stress-Strain curve of Pure PSR.



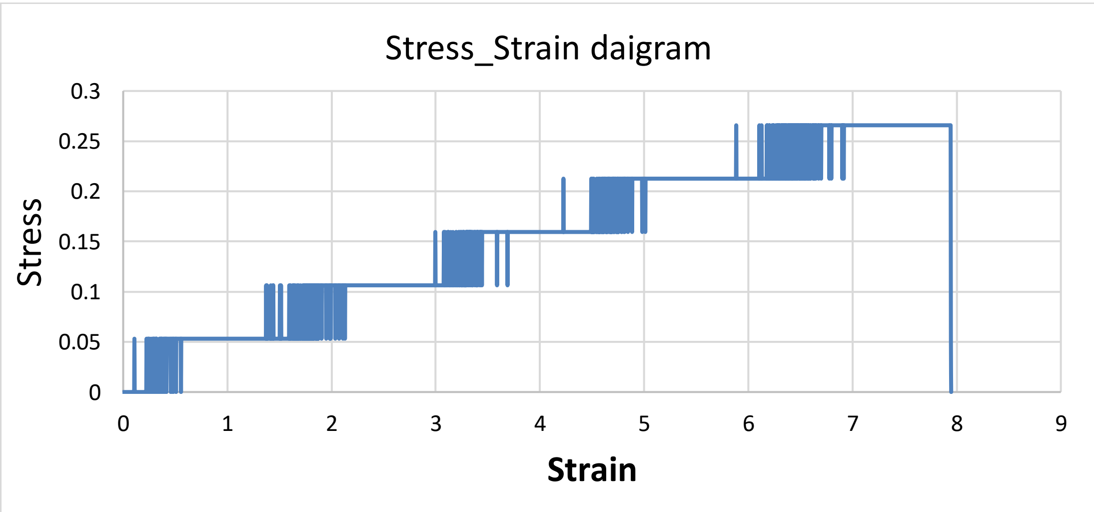
Stress-Strain curve of 3% PSR/C.



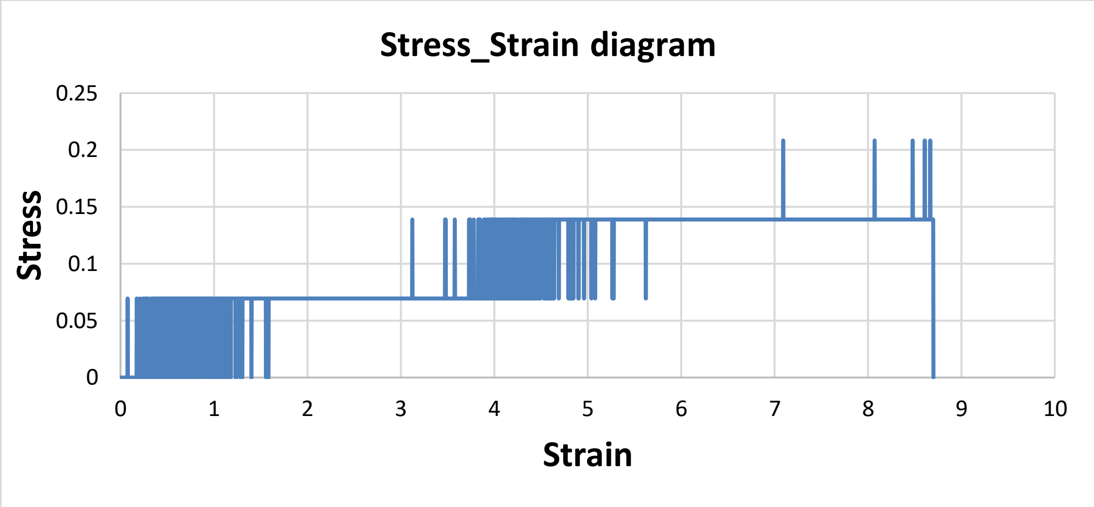
Stress-Strain curve of 3% PSR/W.



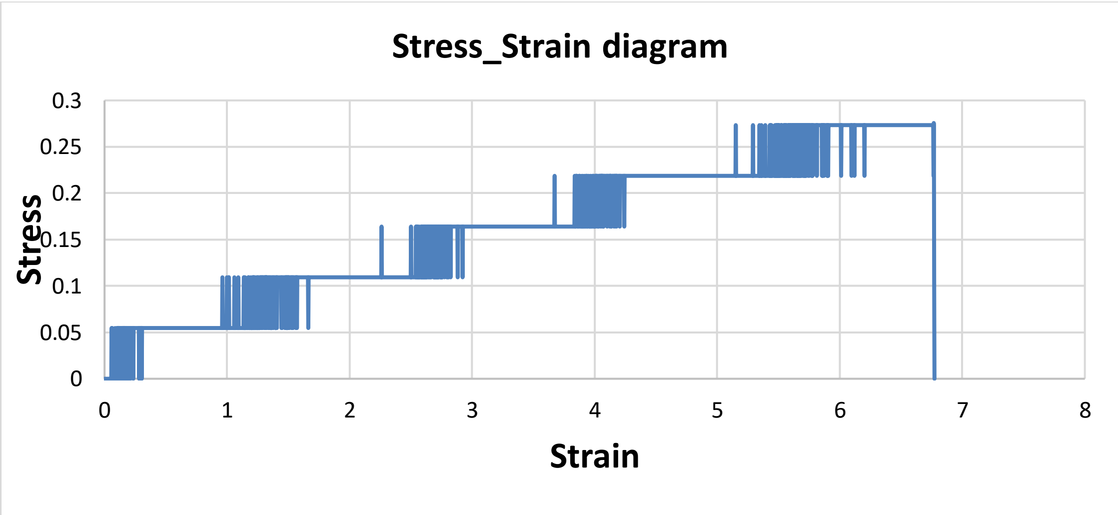
Stress-Strain curve of 6% PSR/C.



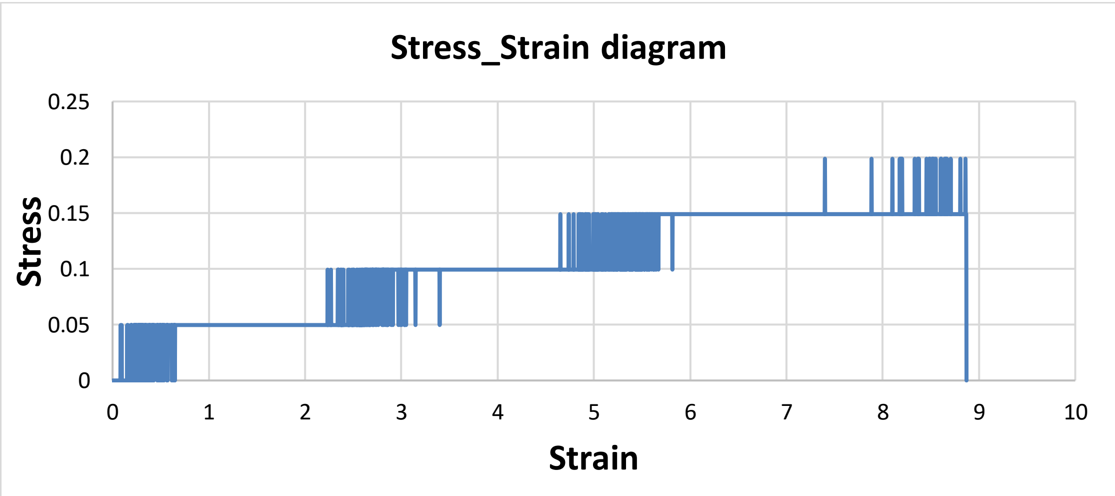
Stress-Strain curve of 6%PSR/W.



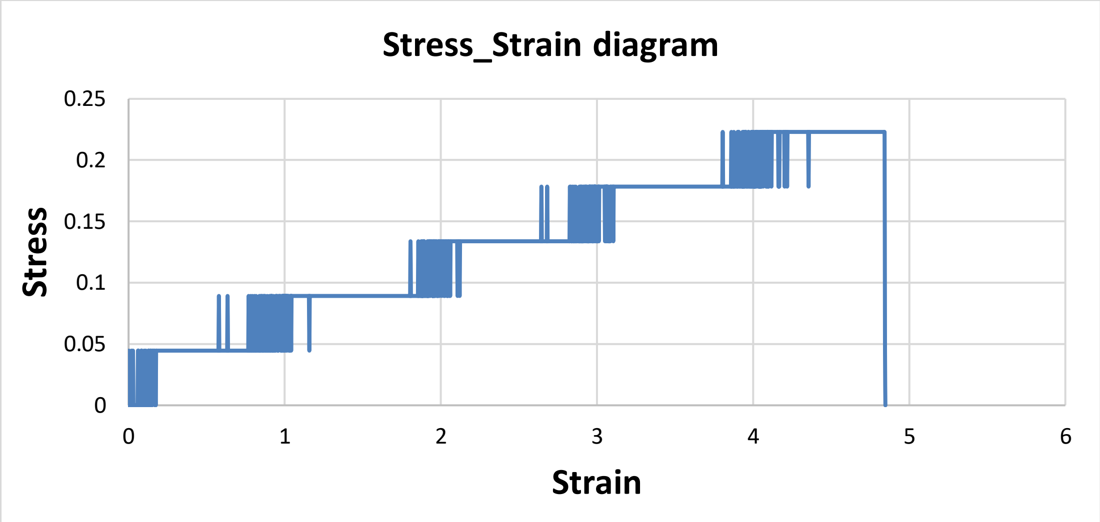
Stress-Strain curve of 9%PSR/C.



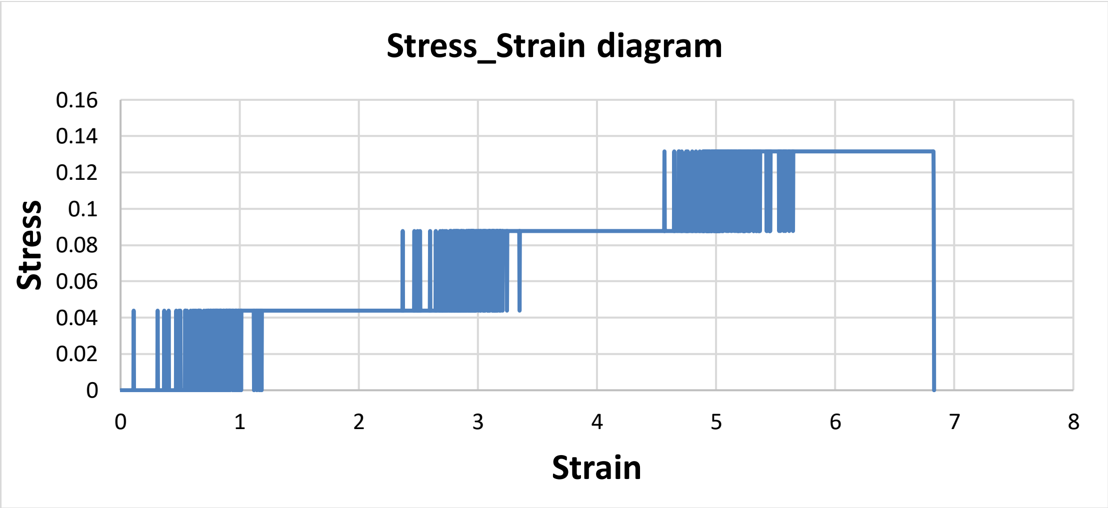
Stress-Strain curve of 9%PSR/W.



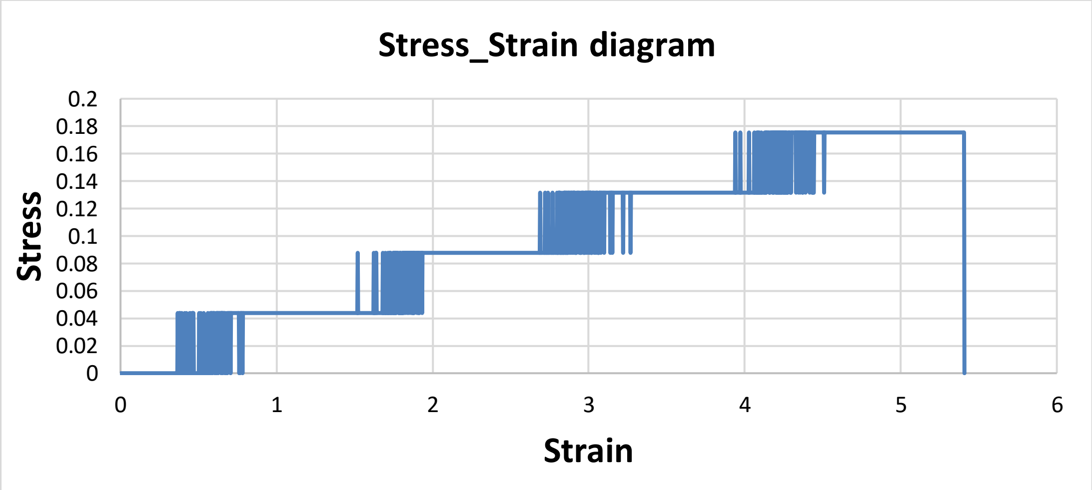
Stress-Strain curve of 12% PSR/C.



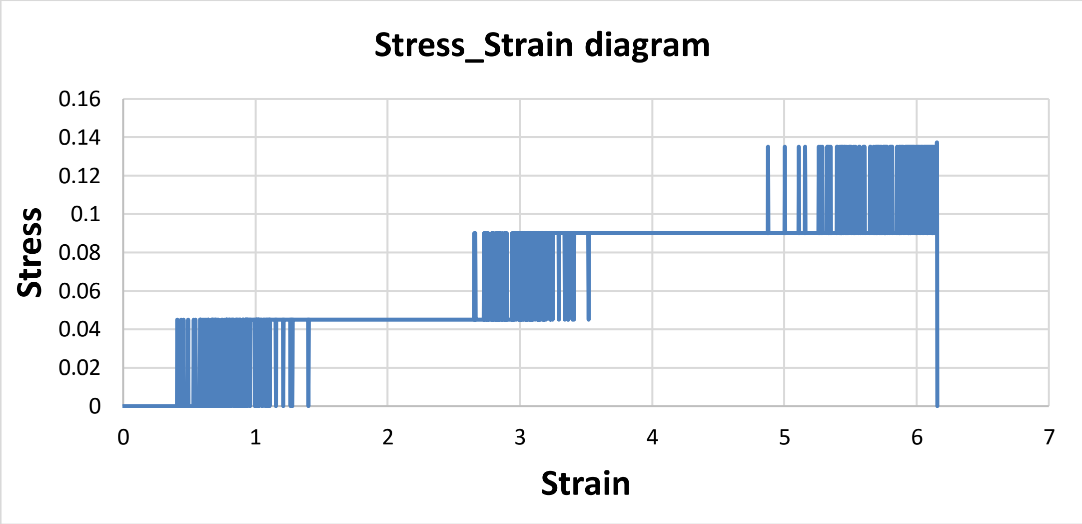
Stress-Strain curve of 12% PSR/W.



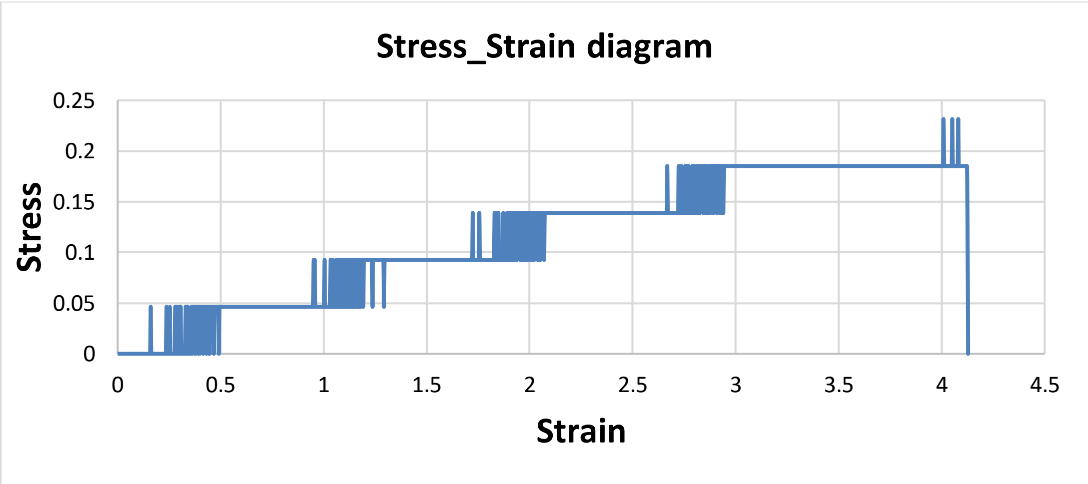
Stress-Strain curve of 15% PSR/C.



Stress-Strain curve of 15% PSR/W.



Stress-Strain curve of 20% PSR/C.



Stress-Strain curve of 20%PSR/W.

الخلاصة

يهتم العالم حاليًا بالحفاظ على البيئة وتقليل التلوث من خلال استخدام النفايات المعاد تدويرها فضلاً عن تقليل التكاليف. إن القوانين البيئية الصارمة وعدم وجود مكبات في المناطق الحضرية تجعل التخلص من نفايات الهدم مشكلة. حيث تعتبر مخلفات الخرسانة من الملوثات الخطرة على البيئة. لذلك فإن إعادة تدوير الخرسانة هي موضوع بحث مهم من أجل منع الاحتباس الحراري. مواد البناء مثل الأسمنت والنفايات الخرسانية تزيد من ظاهرة الاحتباس الحراري ، وهي مشكلة عالمية في الوقت الحاضر ، حيث يؤثر تلوث الخرسانة على الأرض والمحيطات. الكائنات الحية وخاصة الحيوانات البحرية ، يمكن أن تتأثر أيضًا بانبعاثات ثاني أكسيد الكربون في الهواء الذي نتنفسه من الخرسانة المعاد تدويرها ، كما يحافظ على البيئة ، لهذه الأسباب ، هناك حاجة ملحة لإزالة نفايات الخرسانة من البيئة. حيث يتم إضافة الأسمنت البورتلاندي (C) مرة ومخلفات الخرسانة (W) مرة أخرى بنسبة مضافة (0،3، 6، 9، 12 ،15 و 20%) إلى مطاط عديد الكبريتيد (PSR) مع الأخذ في الاعتبار أن المادة اللاصقة تحتفظ خصائص الالتصاق والشد كشيء أساسي موازٍ لأهميته في تقليل التكلفة. تم تحضير المواد المركبة بخلط مطاط عديد الكبريتيد (بنسبة 95٪ بوليمرات: 5٪ مقسى) مع الأسمنت مرة واحدة ونفايات الخرسانة مرة أخرى.

تظهر نتائج(FTIR) أنه لا يوجد تفاعل كيميائي بين عينات مطاط البولي سلفايد والمواد المضافة الأسمنت مرة و مخلفات الخرسانة مرة اخرى لان تداخل الجسيمات هو تداخل فيزيائي.

وأظهرت نتائج اختبار الشد أن قوة الشد القصوى تزداد لمركب البولي سلفايد مع زيادة محتوى مخلفات الخرسانة ، وهي أعلى من مقاومة الشد القصوى لعينات البولي سلفايد النقية ، ولكن عند إضافة الأسمنت إلى البولي سلفايد تزيد مقاومة الشد النهائية للبولي سلفايد المقواه بالأسمنت بشكل طفيف حتى نسبة الإضافة (12٪) (0.199ميكا باسكال) عند مستوى الحد الأقصى لقيمة مقاومة الشد لعينات البولي سلفايد النقية ثم تقل مقاومة الشد بنسبة (15٪ و 20٪).

وأظهرت نتائج الاستطالة أن استطالة المواد المركبة تتناقص مع زيادة كمية إضافة الأسمنت ونفايات الخرسانة. ويكون النقصان أكثر للعينات المقواة بمخلفات الخرسانة أكثر من المقواه بالأسمنت .

أظهرت نتائج اختبار الالتصاق أن قوة الالتصاق تظهر تحسنًا عند إضافة من 3٪ إلى 20٪ من الأسمنت ومخلفات الخرسانة .

وأظهرت نتائج اختبار صلابة (Shore A) أن قيم البولي سلفايد المقوى أعلى منقيمة البولي سلفايد النقي . تظهر صور المجهر الإلكتروني (SEM) تكتلاً في نفايات الخرسانة وسطح خشن وغير منتظم مما ينعكس على الخواص الميكانيكية. يمكن ملاحظة أن شكل نفايات الخرسانة أكثر خشونة وأكبر من الأسمنت وأن جزيئات الأسمنت لها شكل أنعم من مخلفات الخرسانة.

جمهورية العراق

وزارة التعليم العالي والبحث العلمي

جامعة بابل

كلية هندسة المواد

قسم هندسة البوليمر والصناعات البتروكيمياوية

تحضير وتوصيف لواصق بوليمرية لتطبيقات الخرسانة

رسالة

مقدمة الى كلية هندسة المواد/ جامعة بابل وهي جزء من متطلبات نيل درجة الماجستير في هندسة المواد/ البوليمر

من قبل الباحثة

زينب ماجد محمد

(بكالوريوس في هندسة المواد 2018)

بإشراف

أ.د. ذو الفقار كريم مزعل

1443 هـ 2022م