INFLUENCE OF WASTE FOUNDRY SAND (WFS) AND RICE HUSK ASH (RHA) ON THE MECHANICAL PROPERTIES OF POLYMER CONCRETE

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Approval of the Graduate School of Natural and Applied Sciences, Atılım University.

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ABSTRACT

INFLUENCE OF WASTE FOUNDRY SAND (WFS) AND RICE HUSK ASH (RHA) ON THE MECHANICAL PROPERTIES OF POLYMER CONCRETE

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Polymer concrete (PC) is a composite which contains aggregate, filler, bound together with a thermoset resin instead of cement and water. It is used as a repairing material due to its high bonding capability and good mechanical properties. On the other hand, it is quite expensive compared to ordinary concrete. The aim of this study is to investigate the effect of waste materials such as waste foundry sand (WFS) and rice husk ash (RHA) on mechanical properties of PC. RHA is used as a filler, while epoxy is used as a binding material in this study. The tests were conducted according to ASTM standards, where the compressive strength, splitting tensile strength, and flexural strength of the PC specimens were determined with various weight proportions of WFS, epoxy, and RHA. Firstly, five types of mixtures without RHA were prepared to determine the optimum proportion of epoxy and WFS. Secondly, the optimum proportion of RHA was determined for the optimum epoxy/WFS composition. The effect of normal sand (NS) in PC as a control was explored. The weight percent epoxy/WFS ratios were prepared as 10/90, 20/80, 25/75, 30/70 and 40/60, while RHA was tested for 5, 10, and 15% additions by weight. Scanning

Electron Microscopy (SEM) was used to monitor the spatial distribution as well as adhesion between PC components. X-ray fluorescence (XRF) technique was performed to determine the chemical compositions of WFS and NS. Finally, Fourier Transform Infrared Spectroscopy (ATR-FTIR) was used in order to point out the chemical differences between WFS and NS. It was found that the average compressive strength, splitting tensile strength, and flexural strength values were all increased to their maximum for 25/75 epoxy/WFS ratio. On the other hand, the mechanical properties have been noticeably improved with 10% (by weight) RHA addition. Furthermore, using WFS as aggregate leads to better results in both compressive strength and flexural strength values compared to NS.

Keywords: Polymer concrete, Epoxy, Waste foundry sand, Rice husk ash, Mechanical strength, Recycling

ÖZ

ATIK DOKÜM KUMU VE ÇELTİK KÜLÜNÜN POLİMER BETONUN MEKANİK ÖZELLİKLERİNE ETKİSİ

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Polimer beton agrega ve filler malzemenin çimento ve su yerine termoset bir reçine ile bağlanmasıyla oluşan bir yapıdır. Yüksek bağlayıcı özelliği ve iyi mekanik özelliklerinden dolayı onarım malzemesi olarak kullanılmaktadır. Bunların yanı sıra bu malzeme normal betona göre oldukça pahalıdır. Bu çalışmanın amacı atık döküm kumu (ADK) ve çeltik külü (ÇK) gibi atık malzemelerin polimer betonun mekanik özelliklerine olan etkisini incelemektir. Çeltik külünün filler olarak kullanıldığı bu çalışmada epoksi bağlayıcı malzeme olarak kullanılmıştır. ASTM standartlarına göre yapılan testlerde polimer beton numunelerinin basınç, yarmada çekme ve eğilme dayanımları farklı atık döküm kumu, epoksi ve çeltik külü oranları için belirlenmiştir. İlk aşamada çeltik külü kullanılmadan optimum epoksi/atık döküm kumu oranının belirlenmesi için beş farklı karışım hazırlanmıştır. Daha sonra elde edilen optimum karışım oranı için optimum çeltik külü oranı belirlenmiştir. Kontrol için normal kumun polimer betona etkisi de incelenmiştir. Epoksi/atık döküm kumu oranları 10/90, 20/80, 25/75, 30/70 ve 40/60 olarak seçilirken çeltik külü karışım ağırlığının %5, %10 ve %15'i oranlarında kullanılmıştır. Topografik dağılımın izlenmesi ve polimer beton bileşenlerinin birbirleriyle yaptıkları bağları gözlemlemek için taramalı elektron mikroskobu (SEM) kullanılmıştır. Bunlara ek olarak, X ışını floresans tekniği kullanılarak atık döküm kumu ve normal kumun kimyasal özellikleri belirlenmiştir. Son olarak atık döküm kumu ve normal kum araşındaki kimyasal farkları belirlemek amacı ile Fouirer Transform İnfrared Spektrometre (ATR-FTIR) analizleri yapılmıştır. Ortalama basınç, yarmada çekme ve eğilme dayanımlarının hepsinin en yüksek değerlerine 25/75 epoksi/atık döküm kumu oranında ulaştığı tespit edilmiştir. Bunun yanında, söz konusu mekanik özelliklerin %10 çeltik külü eklendiğinde kayda değer bir şekilde iyileştiği görülmüştür. Ayrıca agrega olarak kullanılan atık döküm kumunun normal kuma göre daha yüksek basınç ve eğilme dayanımlarına sahip olduğu belirlenmiştir.

Anahtar Kelimeler: Polimer betonu, Epoksi, Atık döküm kumu, Çeltik külü, Mekanik dayanım.

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LIST OF SYMBOLS

ASTM	American Society for Testing and Material
ATR-FTIR	Attenuated total reflectance-Fourier transform infrared spectroscopy
AMOE	Apparent modulus of elasticity
NS	Normal sand
PC	Polymer concrete
RHA	Rice husk ash
SEM	Scanning electronic microscopy
WC	Water content
WFS	Waste foundry sand
XRF	X-ray fluorescence
d	Depth of the specimen
L	Span length
M1	Mass of the container
M2	Mass of the container and wet sample
M3	Mass of the container and the dry sample
P	Flexural load
W	Width of the specimen
σ	Flexural stress

CHAPTER 1

INTRODUCTION

Concrete is known as a ceramic-based composite material that contains cement, aggregates, water, and additives. Cement combined with water acts as a binding factor, while the aggregate admixture is used as load-bearing components for supporting structure. Concrete has been the most important material in the construction industry for over many decades, due to its high weather resistance, low price, high durability, and good mechanical properties like superior compressive strength. On the contrary, its tensile strength is low and thus steel reinforcement is needed particularly for structural systems. In addition, several disadvantages can also be listed for concrete such as low toughness, which is 1-2% of steel, and long curing time, where compressive strength reaches its peak after 28 days.

As an alternative, polymer concrete (PC) is known to be slightly complex composition which contains aggregates, filler, and a thermoset resin as binding material instead of cement and water. PC was firstly used in the 1950s. It has better properties than ordinary concrete such as high durability, low water impermeability, and high chemical resistance (Hameed and Hamza, 2019). Since PC can reach almost 90% of its strength at the first 7 days, its overall manufacturing time is quite shorter than ordinary concrete which makes it more attractive. On the other hand, its high cost is a big disadvantage. Therefore, using waste materials as ingredients can be a reasonable initiative to reduce the cost of PC.

1.1 Applications of polymer concrete

PC has been used in the construction and manufacturing industry since the 1950's. Due to its favorable properties such as rapid curing, high strength, high chemical resistance, and bonding capability to ordinary concrete, it is mostly used as a repairing material. Moreover, PC is particularly required for machine bases as bedding material where vibration absorption is critical as shown in Fig. 1.1. Another example given in Fig. 1.2 where it is used as a cover coating. Also, utilization of PC as a repairing material for damaged ordinary concrete structures can be seen in Fig.1.3. Furthermore, it can be used in the construction of the railroad crossings as shown in Fig. 1.4. Finally, PC can be utilized as base material for a transformer and a control cabinet vault used for wind power as given in Fig. 1.5. It also can be used as a cladding to ordinary concrete.

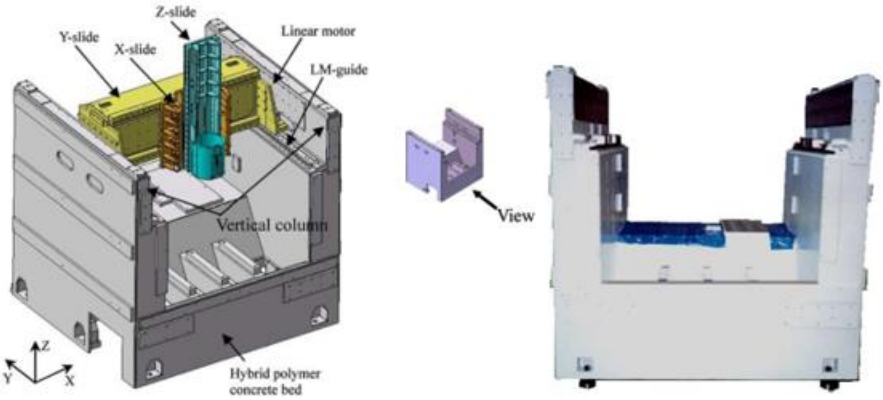

Figure 1.1 Hybrid PC and PC machine tool bed (Lee et al., 2008).

Figure 1.2 (a) The application of PC with epoxy as a cover, and (b) repaired and non- repaired surfaces with PC (Momtazi et al., 2015).

Figure 1.3 PC repair (Fowler, 2007).

(a) (b)

Figure 1.4 (a) Schematic of PC railroad crossing system, (b) Installed PC railroad crossing system (Fowler, 2007).

Figure 1.5 PC base for a transformer and a control cabinet vault used for wind power (Fowler, 2007).

Since waste materials can affect the economy and environment negatively, using them in construction applications was an option to reduce environmental problems. For quite some time, waste landfills have been expanding beyond their borders and leaking out and contaminating our habituates. In other words, recycling and reusing have become an urgent policy adopted for reducing the hazardous effects of waste materials, reducing air pollution, conservation of resources, energy-saving, and creating new employment opportunities (Zhang et al. 2019).That was a motivation to reuse waste foundry sand (WFS) dumped after metal casting processes as an aggregate for PC. Foundry sand is highly used for sand casting processes of ferrous and nonferrous metals. Following the mold knock-out stage, this sand is dumped as piles and rarely reused since it needs a costly pyritization process to burn out the covering resin. However, it contains high-quality silica-sand which can be valuable for other applications. In addition, there are many foundries in Turkey and other countries, which means a huge amount of foundry sand is wasted once they are used.

Likewise, rice husk is another valuable by-product of one of the most popular food. Annually, about 76 million tons of rice have been produced all over the world for the last 10 years. Rice husk is the protective coat of the rice grain and therefore the by-product of rice the milling process which corresponds to about 23% of total rice weight. Rice husks can be utilized as additives for concrete blocks, fuel in power plants, insulating fire bricks, and for production of acids like acetic acid for vinegar production. Rice husk ash (RHA) is the resulting material which remains as soot after burning the rice husk at temperatures around 700 °C. In addition to being an excellent filler to the concrete compositions, RHA has been used in other fields such as ceramic production as well as steel manufacturing (Mistry, 2016).

Thereby, WFS and RHA are two waste materials recycled or reused in the production of PC throughout this study.

1.2 Aim and scope

The aim of this investigation is to use waste materials like WFS as aggregate and RHA as filler in order to develop an environmentally friendly as well as cost-efficient PCs. To evaluate the mechanical properties of this newly developed PC, compressive strength, flexural strength, and splitting tensile strength tests were performed on the

PC samples. Finally, these values were compared with the mechanical properties of the PC produced with normal sand as control.

1.3 Thesis outline

This study consists of five chapters. The first chapter contains the introduction which includes the motivation and general scope of the study. The second chapter contains the literature survey about the previous studies. In the third chapter, the properties of materials that have been used are given and the methodology of the study is introduced. In the fourth chapter, the results of the performed tests are presented. In addition, discussion of results is also done in this part. Finally, in the last chapter, the conclusions and recommendations for future studies are presented.

CHAPTER 2

LITERATURE REVIEW

There are several studies in the literature that investigated the production of PC with or without waste materials. In this chapter, some important research works are summarized.

Gri and Ball (2000) investigated the flexural strength of PC. Fine aggregate silica sand, chopped strand glass fibers, and pigment paste were added into the polyester resin which was used as binder. They reported that the flexural strength of PC was improved when 20% polyester resin by weight was used with 1.5% chopped glass fibers by weight.

Reis and Ferreira (2004) studied the influence of fibers on fracture mechanics of PC. Foundry sand, carbon fiber, and chopped glass fibers were mixed with epoxy resin which was used as the binder. It was found that 29% carbon fiber reinforcement with 13% glass fiber by weight combination enhanced the fracture toughness of PC.

Rebeiz et al. (2004) investigated the use of fly ash as a replacement for sand in PC. Flexural, shear, compressive and tensile bond strength to ordinary concrete, and the creep compliance of the PC material tests were done. Unsaturated polyester resin, fly ash, gravel, steel reinforcement, and sand were used in their study. It was reported that the replacement of sand with fly ash by 15% by weight has enhanced all the mechanical characteristics since better workability can be achieved by fly ash than sand.

Proszek el al. (2004) studied both compressive strength and modulus of elasticity of PC compounds and made a comparative assessment of PC with ordinary concrete. Materials used in that study were fly ash, river sand, and unsaturated polyester resins. It was reported that the addition of fly ash improved both compressive strength and elastic modulus of PC. In addition, they stated that modulus of elasticity values were at acceptable levels compared to ordinary concrete.

Dos Reis (2005) investigated the compressive strength of PC. Compressive strengths were measured to determine the mechanical properties of the samples. Epoxy resin was mixed with foundry sand, carbon fiber, and chopped glass fiber. The presented results showed that 1% addition of glass fiber by weight with 2% carbon fiber reinforcement by weight improved the compressive strength of PC. In addition, PC has shown high compressive strength compared to ordinary concrete.

Tawfik and Eskander (2006) studied the mechanical characterization of PC made by using two types of marble wastes with different sizes, basalt aggregates, and recycled polymer. Compressive and splitting tensile strength tests were determined in their study. Styrenated polyester and two types of filler which are styrenated polyester based on recycled polyethylene terephthalate wastes and marble wastes were used. It was found that using coarser marble wastes showed better results in compressive strength and with increasing methyl ethyl ketone peroxide the compressive strength was improved. On the other hand, tensile strength results obtained from their experiments were acceptable when compared with ordinary concrete.

Reis (2006) studied the mechanical properties of PC reinforced with natural fibers. Epoxy was mixed with foundry sand, and reinforcement fibers such as coconut fiber, sugar cane bagasse, and banana fibers. Flexure and fracture tests have been performed in order to compare the natural reinforced PC, synthetic fiber reinforced PC, and unreinforced PC. It was found that the use of coconut fiber increased the flexural strength compared to both unreinforced and synthetic fiber reinforced PC. On the other hand, the use of both sugar cane bagasse or banana fiber was reported to decrease the flexural strength.

Bărbuţă and Lepădatu (2008) investigated PC made from epoxy resin as binder, silica fume as filler, and aggregates. Compressive, flexural, splitting tensile and adhesion strength tests were used to determine the mechanical properties of PC. The used percentages of polymer started from 12.4 to 18.8% by weight. It was found that the optimum proportion of PC was about 15.6% by weight.

Jo and Park (2008) studied the compressive strength and splitting tensile strength of PC. Polyester resin was mixed with recycled poly ethylene terephthalate plastic waste, recycled concrete aggregates, and calcium carbonate ($CaCO_3$) as filler. It was reported that by the addition of recycled aggregates, the compressive strength and splitting tensile strength of PC were all improved and reached their highest value at 13% resin content.

Bărbută et al. (2010) compared the mechanical properties of PC with different types of filler materials. Compressive, splitting tensile and flexural strength tests were done. They used materials were aggregate, the silica fume and fly ash as fillers in epoxy resin. It was reported that using fly ash as filler leads to better results in both compressive and flexural strength compared to silica fume. The addition of silica fume and fly ash by 10.4% and 12.8% by weight, respectively, have enhanced both compressive and splitting tensile strength while decreased the flexural strength.

Agavriloaie et al. (2012) investigated both the mechanical and thermophysical characterization of epoxy polyurethane acryl polymer concrete. Epoxy and crushed granite were used in their study and compressive strength, pullout strength, flexural strength, and elasticity modulus were obtained by several mechanical tests. On the other hand, thermo-physical properties such as thermal shock strength, bulk density in natural and dry state, thermal conductivity, relative and absolute mass humidity, linear thermal dilatation, chemical resistance, frost-thaw resistance as well as water adsorption resistance were also studied. In the study, the high performance of epoxy polyurethane acryl polymer concrete with light weight was observed, and it was reported that epoxy polyurethane acryl polymer concrete could be used in industrial buildings where high chemical and thermal resistance are highly needed.

Shi-cong and Chi-sun (2013) investigated PC mixtures prepared by using fly ash, metakaolin, and recycled glass as aggregate. The materials were prepared according to the weight factors. Water absorption amount, compressive strength, flexural strength and modulus of elasticity were obtained during the experiments. It was found that the addition of both metakaolin and fly ash had significantly enhanced the flexural and compressive strength, and elastic modulus of the PC. Furthermore, it was reported that PC which was made of metakaolin, waste glass and fly ash combination has low apparent porosity as well as water absorption level. It was also reported that the addition of metakaolin and fly ash with recycled glass enhanced the mechanical properties of PC overall.

Lokuge and Aravinthan (2013) studied the effect of fly ash on the behavior of PC with different types of resin. Polyester, vinyl ester and epoxy resin were used as binder with class F fly ash as filler and sand. Compressive, splitting tensile, and flexural strength tests were performed. It was found that the addition of fly ash decreased the amount of resin used and increased the compressive strength, while decreasing the fly ash amount influenced ductility positively. On the other hand, it was observed that the modulus of elasticity increased with increasing fly ash content, while increasing the amount of fly ash caused a decline in splitting tensile and flexural strength.

Jung et al. (2014) investigated the PC as a repairing material in runways. The material properties that were measured in the study were compressive and flexural strength, Young's modulus, the coefficient of thermal expansion, and mass density. Four epoxy curing ratios of 10, 15, 20, and 30% by weight were used in their study. It was observed that compressive strength, Young's modulus, and mass density were enhanced at 20% epoxy composition, meanwhile, flexural strength was improved at 30% epoxy composition. Also, it was shown that 6 hours is needed for the curing of the resin, which means PC can be a fast choice for runway repairing. It was also noted that PC's thermal expansion coefficient is two and half fold higher than ordinary concrete.

Niaki et al. (2018) studied the mechanical and thermal properties of PC. PC was composed of epoxy as a binder, basalt aggregate, chopped basalt as fiber and class F ultrafine fly ash as filler, together with nano-clay particles as reinforcing agents. Compressive, flexural, splitting tensile and impact strength values were measured and the effect of various temperatures up to 250 °C on mechanical characteristics of fiber reinforced PC were studied. It was found that the mechanical properties and thermal conductivity were improved with the addition of basalt fiber. Furthermore, nano-clay addition increased the value of thermal conductivity and all mechanical properties with the exception of splitting tensile strength.

Niaki et al. (2018) also studied the mechanical properties of epoxy/basalt PC. Splitting tensile, compressive and flexural strength were measured in their study. Crashed basalt was used as aggregate and epoxy resin was used as binder. It was reported that the mechanical properties of PC were reached their highest value at 25% epoxy resin. Moreover, they analyzed the effect of temperature and determined that with increasing temperature, mechanical properties of PC deteriorated. They also concluded that the size of aggregate plays an important role in the mechanical properties of PC. For example, using large sized aggregates leads to higher compressive strength, yet lower splitting tensile and flexural strength.

Jafari et al. (2018) investigated the mechanical properties of PC. Materials used were epoxy resin, fine aggregates, and two types of coarse aggregates. 10, 12 and 14% by weight epoxy ratios by weight were used throughout the experiments. Compressive and splitting tensile strength tests were performed at three different temperatures. It was found that increasing epoxy content while decreasing temperature using coarse aggregates increased the mechanical properties.

Sosoi et al. (2018) studied the use of waste materials like sawdust and chopped polyethylene terephthalate as aggregates in PC made by epoxy resin with fly ash. Both density and compressive strength values were determined in this investigation. The results showed that PC with its density around 2.12 g/m^3 which is quite lighter than ordinary concrete. It was reported that using both sawdust and chopped polyethylene terephthalate improved compressive strength of PC, hence waste materials could be a good replacement for aggregates.

Hameed and Hamza (2019) investigated the mechanical properties of PC made up of aggregates from waste materials and compared these values with the ordinary concrete. Polyester resin was used as binder, and concrete debris, ceramic tiles and building block waste, natural and river sand were used as aggregates. Flexural, compressive and splitting tensile strength were performed. It was found that increasing the proportion of resin improves all the values of mechanical properties and the resulting PC had shown better characteristics such as compressive, splitting tensile, and flexural strength comparing to ordinary concrete.

Wang et al. (2019) investigated the mechanical properties and durability of epoxy-based PC modified with rubber crumbs. In this investigation, epoxy resin was mixed with basalt fiber and waste tire rubber particles. The mechanical tests were performed to measure splitting tensile, compressive and interface bond strength of PCs. They found that the addition of 5% solid rubber by weight enhanced both compressive and split tensile strength of PC by 9% and 5%, respectively. On the other hand, it was reduced the thermal conductivity by around 3%.

CHAPTER 3

MATERIALS AND METHODOLOGY

3.1 Introduction

This study is focused on the production of epoxy-based PC by using waste foundry sand and rice husk ash. In order to determine the effects of the waste materials on the mechanical properties of PC, some mechanical tests were performed. In addition, scanning electron micrographs were taken to investigate the adhesion behavior of the PC additives. Furthermore, X-ray fluorescence and attenuated total reflectance-Fourier transform infrared spectroscopy tests were done on these materials to determine their chemical compositions.

Detailed information about the materials used in PC production is given and test procedures and sample preparations are explained thoroughly throughout this chapter.

3.2 Materials

3.2.1 Epoxy

Epoxy is considered as a material that is formed by two groups of epoxides that goes through cross-linking reaction by the use of an oligomeric curing agent. The curing agent can be an amine or a diacid compound. Different types of such resins available on the market come with a monograph. The reaction of olefins with peroxybenzoic acid to epoxides had been discovered by N. Prileschajew in 1909 (Fink, 2005). In 1939 a polymeric material had been produced by using both amines and multifunctional epoxides by Schlack (Fink, 2005). Furthermore, the preparation of bisphenol A diglycidyl was determined in the search for dental materials by Castan (Fink, 2005). Epoxy resins took their place in global markets since 1947 for use in some applications like coating. The reaction of epoxides is limited and is related to the material classes used. The curing agents used to start cross-linking reactions can be classified into three main categories, which are compounds with active hydrogens, ionic initiators, and hydroxyl coupling agents. The curing reaction is polyaddition initially. Therefore, the curing of glycidyl can be achieved particularly by amines and another nitrogen-containing compound (polyamides). Since amines could enhance the curing process at room temperature, the prosses is known as cold curing. The reaction of the glycidyl group with an amine and with a hydroxy group can be shown in Fig. 3.1 (Fink, 2005).

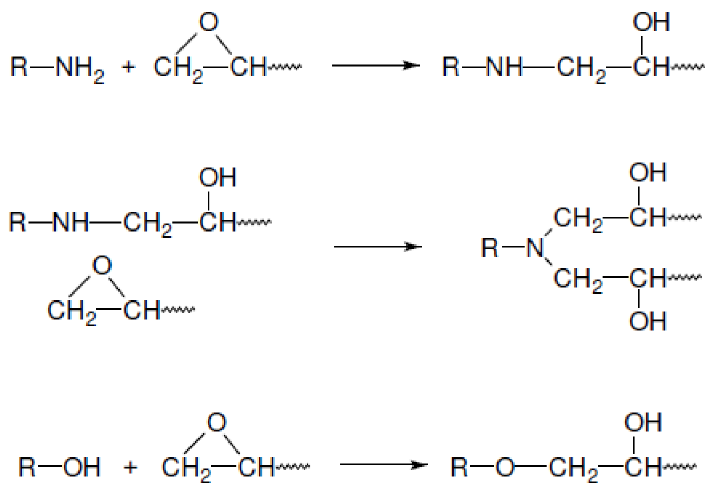

Figure 3.1 Reaction of the Glycidyl group with an amine and with a Hydroxy group.

3.2.1.1 Thermal curing

Isothermal curing can assess the highest thermal stability of the polymers. According to many experimental trials with respect to temperature application mode, a higher transition temperature and superior thermal stability can be accomplished by a slower heating rate. The type of the resin plays an important role in enhancing the mechanical properties of the PC. The characteristics of polymeric resin can provide enhancement on adhesion strength, chemical and thermal resistance, and corrosion resistance. Also, due to its high binding characteristic, epoxy is considered as a strong holder under pressure (Fink, 2005).

In this study, Av75 type epoxy provided by Duratek was used in the production of PC (Fig. 3.2). This epoxy-based resin is solvent-free. This resin has high bounding capability with enhanced adhesion characteristics as well as good chemical resistance. Some properties of Av75 are given in Table 3.1.

Figure 3.2 Duratek Av75 epoxy (A 65%, and B 35%) .

Table 3.1 Properties of Duratek Av75 type epoxy.

Density	1.09 ± 0.05 g/cm^3
Viscosity (at room temperature)	1000 ± 200 mPa.s
Water absorption and permeability	< 0.1 kg/m^2 .h$^{0.5}$
Impact toughness	≥ 4 Nm

3.2.2 Foundry sand

Foundry sand is a high-quality silica sand and used for the construction of the molds used for sand casting of ferrous and nonferrous metals. Because of its high thermal conductivity, it has been used as a molding material for ages. Foundry sand goes through many preparation processes to attain robustness and strong structure by addition of several binders and additives so that it can bear hot metal flow. It is classified according to additive or binder used. For example, if clay is used as a binding material in the sand, it is called green bonded sand and if chemical binders are used, it is called chemically bonded sand. Therefore, the main components of green foundry sand are silica sand and bentonite. During mold preparation, generally, 85-95% by weight silica sand is mixed with 4-10% by weight bentonite. The percentages of carbonaceous additives could be ranged 2-10% by weight, in order to enhance the casting surface. It also consists of oxides such as MgO, K_2O, TiO_2. On the other hand, the composition of chemically bonded foundry sand mainly consists of silica sand and chemical binders. In this case, 93-99% by weight silica sand is mixed with 1-3% by weight chemical binders. Chemically bonded sand mixture is prepared by adding the chemicals into the sand which are mixed thoroughly in order to have an effective reaction that cures and hardens the mold mass throughout its all structure. The epoxy resins are the most common chemical binders that have been used in casting sand. With the addition of carbonaceous fillers, the color of the green foundry sand is converted into black while in case of chemical additives the foundry sand gets a light color. Green foundry sand is used bountifully in molding operations while chemically bonded foundry sand is used both in mold and core making processes (Bavita Bhardwaj, 2017).

3.2.3 Waste foundry sand

Waste foundry sand (WFS) is considered as a waste product of sand-casting process of ferrous and non-ferrous metals. Even though it is usually dumped as waste, it is still a high-quality silica sand. The molding sand can be recycled repeatedly during the casting process. After some number of cycles, the recycled sand cannot be used any further in casting operations. Then the sand gets dumped as a waste material and no longer be used in any casting operations. The main waste product of the foundry industry is WFS and it is disposed of by dumping at a landfill site. However, due to the rising disposal costs, landfills are becoming another problem. There are about 3000 foundries in the US alone which use around 100 million tons of sand with generated waste sand by approximately 6-10 million metric tons annually. India which is considered as the third biggest casting manufacturer after the US and China produce approximately 15 million metric tons of waste sand annually. Due to the large pile-ups of foundry sand in landfills, researchers seek ways to reuse this sand-like utilizing in concrete as a replacement of fine aggregates (Bavita Bhardwaj, 2017).

WFS which is used in this study was provided by Derman Döküm Industries Ltd., Ankara, Turkey (Fig. 3.3). Since sand can hold humidity, WFS was oven-dried in Elektro-mag M6040P oven (Fig. 3.5) for 24 hours at 105 ℃ according to ASTM standards (ASTM D2974). Then it was cooled for another 24 hours in the oven at room temperature. WFS was also subjected to sieve analysis to determine its particle size distribution, since it is a waste material consisting of sand particles with different sizes.

Figure 3.3 Waste foundry sand.

3.2.3.1 Sieve analysis

WFS passed from sieve number 8 which means that the particles have diameter less than 2.38 mm. This test was carried out according to the ASTM C33 standard in order to determine the graduation for both WFS and NS. The sieves that are used in the sieve analysis were #8, #16, #30, #50, #100 and #200 which has 2.38, 1.19, 0.6, 0.3, 0.15, and 0.074 mm aperture openings, respectively. The photo of the sieves used in the analysis is shown in Fig. 3.4.

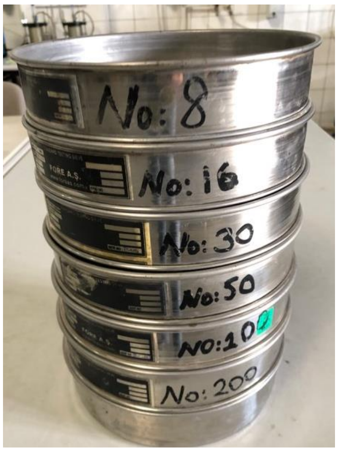

Figure 3.4 Sieves used in the analysis.

Figure 3.5 Elektro-mag M6040P drying oven.

3.2.4 Normal sand

Normal sand (NS) was also used in this study as control for comparison in order to assess the role of WFS in enhancing the mechanical properties of PC. NS used in this study shown in Fig. 3.6 was obtained from one of the aggregate plants in Ankara.

Figure 3.6 Normal sand.

3.2.5 Rice husk ash

Rice is considered as one of the most consumed food in the world. The production of rice has increased particularly in the last decade and reach approximately 760 million tons. Rice husk is the shell surrounding the rice grain and obtained as organic waste following the milling process of rice. It constitutes 20-23% of rice weight, which means more than 150 million tons of rice husk is produced annually as a by-product of rice harvest. Since it can have an adverse effect on the environment, researchers have studied intensely in order to find a value-added application area. Moreover, some other treatments are required on fly ash in order to use it as an eco-friendly material. Also, burning temperature plays a pivotal role in the chemical composition and structure formation. RHA has shown a better effect on the construction materials such as concrete due to its high pozzolanic reaction. When some amount of cement was replaced by RHA in concrete, it improved the characteristics of the concrete without any detectable reduction of strength or durability. When RHA is subjected to a temperature around 500-700 °C, it becomes useable in construction applications (Tuan et al. 2011). In Fig. 3.7, the photos of rice husk and rice husk ash are given. In this investigation, RHA was used in PC composition as filler so as to improve the mechanical properties by filling possible voids in PC structure. RHA was produced by burning rice husk at a controlled temperature up to 600 °C. Oven used for burning is PLF Series 110-130-protherm furnaces (Fig. 3.8). Following the burning process, the weight of RHA was reduced by 75%. The ash was then subjected to compaction by grinding. RHA that was used in PC was passed from sieve #200 which corresponds to particle diameter less than 75µm.

Figure 3.7 Rice husk ash before burning and after burning.

Figure 3.8 PLF Series 110-130 -protherm furnaces.

3.3 Water content

The initial water content of WFS was measured by a test performed according to the ASTM D2216. The procedures were carried out according to the instructions within the code. The weight of the empty container was recorded first to measure the net weight. Then the weight of the container with the sand was measured before keeping it in the oven at 105 ºC for 24 hours. Following drying out procedure, the weight of the sand was measured after 15 minutes halt for it to cool down. The water content was calculated by using the following equation:

$$\mathrm{WC} = \frac{(\mathrm{M2}-\mathrm{M3})}{(\mathrm{M3}-\mathrm{M1})} \times 100 \qquad \textbf{(3.1)}$$

Here, WC is water content, M1 is the mass of the container, M2 is the mass of the container and wet sample, and M3 is the mass of the container and the dry sample.

3.4 Mixture specifications

In this study, different mixtures were prepared in order to obtain the optimum proportion. Firstly, the WFS was placed in the oven ’Elektro-mag-m-6040-p’ for 24 hours to obtain an oven-dry WFS as described above. After it was cooled for another

24 hours in the oven at room temperature, it was used in the PC mixture. The first mixture set was consisted of epoxy by proportions of 10, 20, 25, 30, and 40% by weight and WFS by 90, 80, 75, 70, and 60% by weight, respectively. These proportions were used to obtain the optimum percentage of the epoxy resin with respect to the mechanical properties. Epoxy resin was mixed thoroughly with its hardening agent in 65 to 35% proportion. After that, the WFS was added into the mixture. The mixing process at that stage was done by using a RYOBI EID600RS mixing machine as shown in Fig. 3.9. During the mixing process, some spots were not mixed properly due to the difficulties to reach all the places in the cup, therefore manual mixing was also used where and when necessary to have a homogenous admixture. The second mixture set was prepared after obtaining the optimum ratio of WFS/epoxy which is 3:1. Then this composition was mixed by adding 5, 10, and 15% by weight of RHA while maintaining the optimum ratio between epoxy and WFS. This procedure was done in order to determine the optimum percentage of RHA. While preparing the final composition, first epoxy and its hardener were mixed thoroughly, then WFS and RHA were mixed by hand. Finally, both mixtures were mixed together. The mixing process at that stage was done by using the mixing machine.

Figure 3.9 RYOBI EID600RS mixing machine.

3.5 Mix proportions by weight percentage

Table 3.2 shows the amount of change in % epoxy binder with respect to % WFS addition by weight in the first set of experiments where RHA was not used. The optimum percentage of epoxy was found as 25% (PC 3) which means that 3:1 WFS to epoxy ratio was used for the rest of the experiments. In Table 3.3 the mixture composition used for the second set of experiments are shown. The percentages of the filler material (RHA) was added to the mixture by 5, 10, and 15%, respectively in order to determine the optimum percentage of RHA. It should be noted that the 3:1 ratio between the WFS and the epoxy was kept constant for this set. Finally, the third set, where the mixture proportions used for the control experiments with NS was given in Table 3.4.

Table 3.2 Components of PC with WFS.

Sample	PC1	PC2	PC3	PC4	PC5
Epoxy (%)	10	20	25	30	40
WFS (%)	90	80	75	70	60

Table 3.3 Components of PC with RHA and WFS.

Sample	PC3	PC6	PC7	PC8
Epoxy (%)	25	23.75	22.5	21.25
WFS (%)	75	71.25	67.5	63.75
RHA (%)	0	5	10	15

Table 3.4 Components PC with RHA and NS.

Sample	PC9	PC10	PC11	PC12
Epoxy (%)	25	23.75	22.5	21.25
NS (%)	75	71.25	67.5	63.75
RHA (%)	0	5	10	15

3.6 Mechanical tests

3.6.1 Test specimens

The specimens with the given dimensions were prepared according to the American Standard for Testing and Material (ASTM). Mold materials were chosen carefully to avoid sticking on the mold walls and make the mold release process as gentle as possible without damaging the specimens. Therefore, Teflon was chosen as the proper mold material because of its non-sticking properties. In order to enhance its non-sticking properties and alleviate any possible hassle of releasing the specimens from the mold cavity, sunflower oil was used to coat the mold walls before pouring the mixture. Cylindrical specimens used for both compressive and splitting tensile strength tests with dimensions of 100×50 mm according to ASTM C39 and ASTM C496, respectively as shown in Fig. 3.10. For flexural strength test, prismatic beam specimens with dimensions of 254×25×25 mm according to ASTM C580 was used as shown in Fig. 3.11. A summary of test types, related standards, shapes, dimensions, and number of specimens are given in Table 3.5. The details of these experiments are explained in the next section.

Table 3.5 Mechanical test details.

Test	ASTM standard	Shape	Dimensions (mm)	Number of specimens
Compressive strength	C39	Cylinder	100×50	3
Splitting tensile strength	C496	Cylinder	100×50	3
Flexural strength	C580	Prismatic beam	254×25×25	3

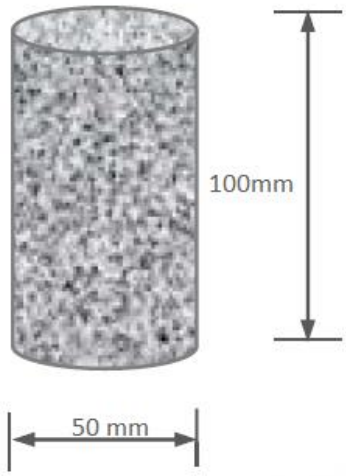

Figure 3.10 Compressive strength and splitting tensile strength test specimens.

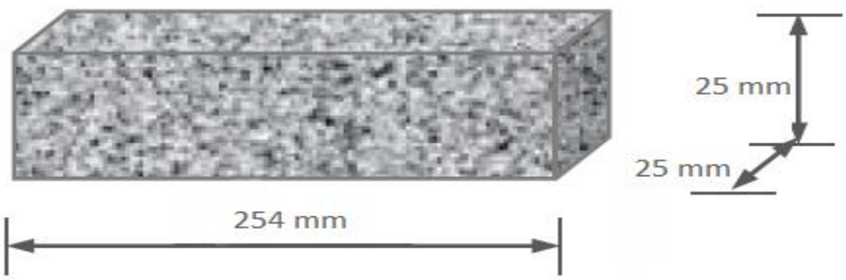

Figure 3.11 Flexural strength test specimens.

3.7 Methodology

In this work, samples prepared according to appropriate ASTM standards were used for compressive, splitting tensile and flexural strength tests to determine the optimum proportions of epoxy and RHA, respectively. All the tests were performed by using Zwick Roell Z300 machine as shown in Fig. 3.12. In these tests, three samples were prepared for each mixture composition. The first set of tests was performed to determine the optimum percentage of epoxy with WFS. The second set of tests were done in order to determine the optimum percentage of RHA for the optimum WFS/epoxy ratio. The last set of experiments was performed to investigate the mechanical properties of PC with the addition of NS with the same proportions of

WFS as control. This comparison would provide a clear vision of the enhancement in the mechanical properties of PC as a function of WFS.

Figure 3.12 Zwick Roell Z300 machine.

3.7.1 Compressive strength

Compressive strength is known as the capability of the material to carry the loads which are subjected on its surface in a pressing manner without any deflection or cracks. Many factors can affect the compressive strength such as polymer-aggregate ratio, polymer strength, quality of polymer, size of aggregate, and quality control measures during the production of PC. According to ASTM C39, compressive strength tests were performed with 1 mm/min crosshead speed with a pressing continuous load over the surface of PC cylinder specimen till the moment of failure. PC specimens were tested 7 days later after casting and curing stage in order to attain 97% of its strength (Bedi, Chandra, & Singh, 2013). Compressive strength values are calculated by dividing the maximum load over the cross-sectional area which is taken as 1963.5 mm^2 in this study. Three specimens were tested for each composition and the average ultimate strength values were obtained with their standard deviations. A moment during a compressive strength test can be seen in Fig. 3.13.

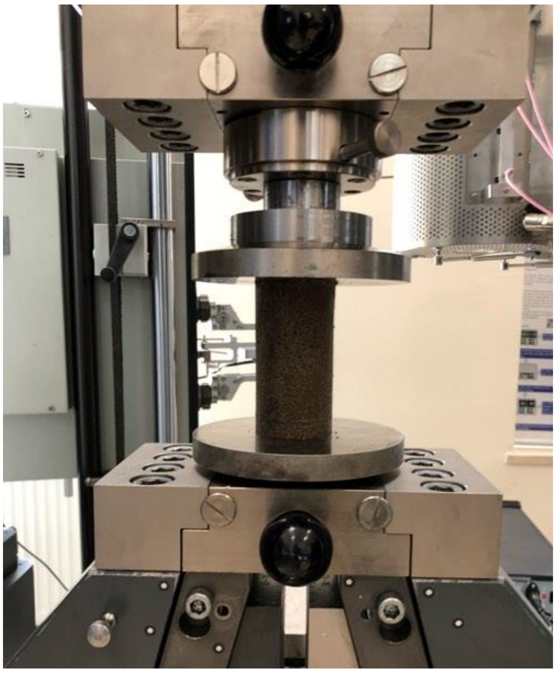

Figure 3.13 Compressive strength test.

3.7.2 Splitting tensile strength

Since tensile strength is quite important to understand the crack propagation manner, splitting tensile tests are performed for the specimens. According to ASTM C496, splitting tensile strength tests were performed with a 0.5 mm/min crosshead velocity and with continuous load over the surface of PC cylinder specimen till the moment of failure. Splitting tensile strength value was calculated by dividing the maximum load over the cross-sectional area which is determined as 5000 mm^2 for this specimen dimensions in this study. Three specimens were tested for each mixture and the average ultimate strength value was obtained. A moment during a splitting tensile strength test is shown in Fig. 3.14.

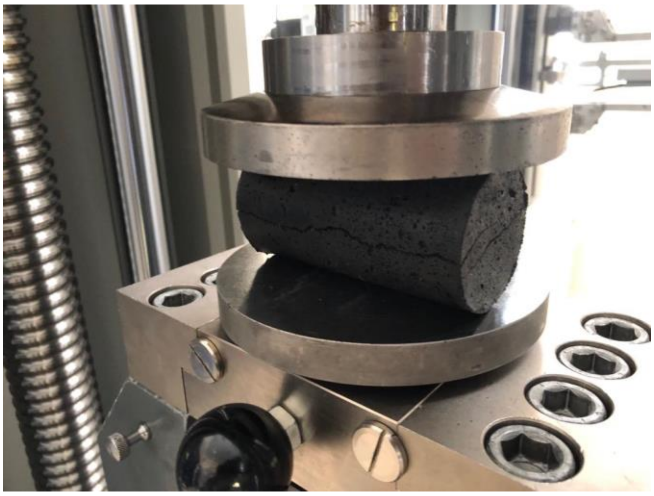

Figure 3.14 Splitting tensile strength test.

3.7.3 Flexural strength

Three-point bending test was performed for the PC specimens according to ASTM C580. The prismatic beams were located on the two supports and the load was applied on the midpoint of the beam. Flexural strength was calculated according to the following equation:

$$\sigma = \frac{3PL}{2wd^2} \tag{3.2}$$

Here, σ is the flexural stress, P is the maximum flexural load applied by the testing machine, L is the span length, w is the width of the specimen, and d is the depth of the specimen. A photograph demonstrating the flexural strength test is given in Fig. 3.15.

Figure 3.15 Flexural strength test.

3.8 Modulus of elasticity

Modulus of elasticity is known as the property which is defined as the ratio of stress to strain within the elastic range. It can be influenced by many factors such as materials used, porosity of the aggregate, loading conditions, stiffness of the material, and the volume fraction of the aggregates (Vakhshouri and Asce, 2018). In this study, since the compliance values of the plates holding the samples under compression were not measured, modulus of elasticity values are not exact. Hence, apparent modulus of elasticity (AMOE) values were calculated accordingly.

3.9 Scanning electronic microscope

Scanning electronic microscope (SEM) is an instrument which focuses a beam of electrons with electromagnetic lenses to raster the specimen surfaces and to get high resolution images at high magnifications like 20000X. Therefore, SEM was used to analyze the microstructure and failure modes of the PC samples and thus to understand the efficacy of the bonding processes between epoxy resin and the other materials. In this study, FEI Quanta 400F Field Emission electron microscope was used as shown in Fig. 3.16.

Figure 3.16 Quanata 400F Field Emission electron microscope.

3.10 X-ray fluorescence

X-ray fluorescence (XRF) is known as an analytical technique which is used to find and determine the elemental composition of materials. XRF test method is performed by exciting the sample by primary X-ray source and measuring the fluorescent (or secondary) X-ray which emanates from the tested sample. This test was done in order to determine and compare the chemical compositions of WFS, NS and RHA. As a matter of fact, the knowledge of the chemical compositions of the mixture components provides a clear image about the elements that affect the adhesive and thus mechanical strengths.

3.11 Attenuated total reflectance-Fourier transform infrared spectroscopy

Attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR) is known as an approach to determine the fingerprint regions of specific chemical groups in polymeric, organic, and inorganic materials. FTIR analysis procedure is achieved by scanning test samples using infrared (IR) light to determine motions of some specific chemical groups. The equipment that was used in this test is the BRUKER ALPHA-PLATINUM-ATR FTIR spectrometer as seen in Fig. 3.17.

Figure 3.17 BRUKER ALPHA-PLATINUM-ATR FTIR spectrometer.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

As stated in the previous chapter, mechanical tests such as compressive, splitting tensile, and flexural strength tests were performed in order to determine the optimum proportions of epoxy and RHA in the PC mixture. In this chapter, the results of these experiments are presented and discussed. In addition, the mechanical properties of PC made with WFS are compared with the one made with NS. SEM, XRF and ATR-FTIR results are also presented in the chapter.

4.2 Test on WFS

4.2.1 Sieve analysis

The test was carried out according to the ASTM C33 standard in order to determine the gradation of the WFS used in the PC mixture. The gradation curve obtained after sieve analysis is given in Fig. 4.1. Same gradation curve is applied for NS used for control experiments.

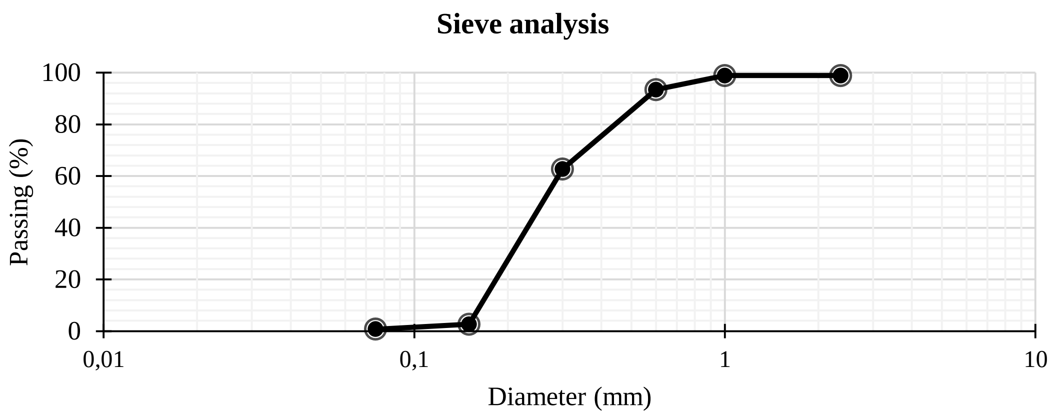

Figure 4.1 Gradation curve for WFS.

4.2.2 Water content

This test was done for the WFS and NS according to the ASTM D2216 standard in order to determine the initial water content. Water content obtained for both NS and WFS are shown in Table 4.1. Even though WFS was dumped outside as a pile and open to atmosphere, it did not absorb water as much as NS according to these results. Most probably carbonized resin coating around the sand grains gave the WFS relative hydrophobicity compared to NS.

Table 4. 1 Water content for both NS and WFS.

Sand	NS	WFS
Water content (%)	0.92	0.20

4.3 Mechanical properties of PC

Compressive, splitting tensile, and flexural strength of PC samples with different epoxy content were obtained using Zwick Roell z300 machine. As stated before, in the first set of experiments 10, 20, 25, 30, and 40% by weight epoxy with corresponding WFS ratios were used, and an optimum content of epoxy for PC was obtained according to the mechanical test results. Then a set of samples were prepared for this optimum ratio with 5, 10 and 15% of RHA so as to determine the final optimum composition of PC with RHA filler. Specimens prepared for the experiments are shown in Fig. 4.2.

Figure 4.2 Specimens prepared for the experiments.

4.3.1 The effect of epoxy amount on mechanical properties of PC

4.3.1.1 Compressive strength

The compressive strength test results are shown in Fig. 4.3 for different epoxy contents. As stated before, three tests for each percentage was carried out. The average values of compressive strength for 10, 20, 25, 30, and 40% of epoxy by weight were obtained as 19.21, 27.04, 30.70, 23.49 and 20.06 MPa, respectively. As seen from these results, the average compressive strength values reach their maximum when the epoxy percentage is 25%. The maximum values of compressive strengths were used in calculations. Fig. 4.4 shows the change in average compressive strength values for different percentages of epoxy in PC. Compressive strength and AMOE values of PC samples for given percentages of epoxy are listed in Table 4.2 and Table 4.3, respectively.

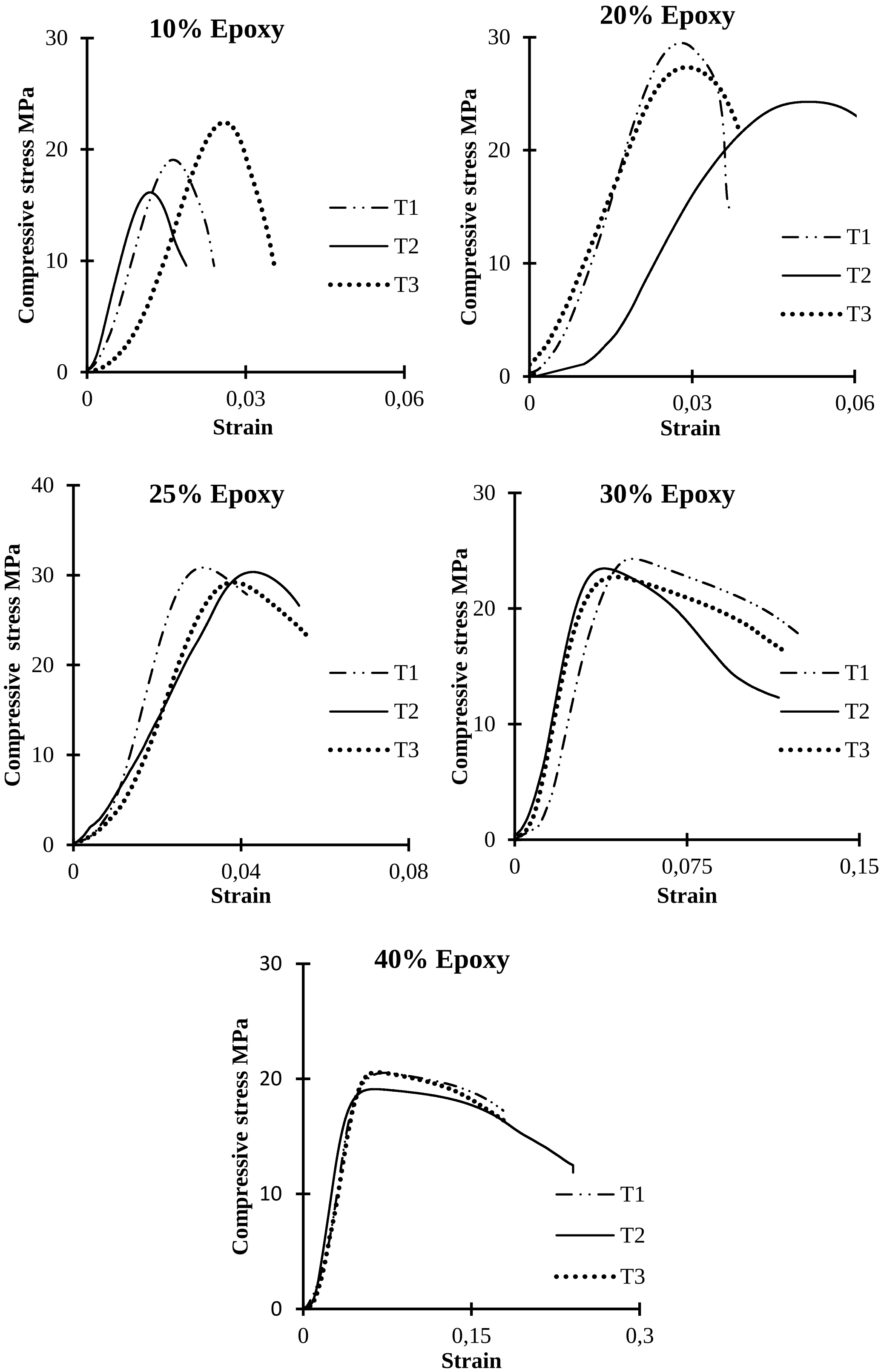

Figure 4.3 Compressive strength test results for (a) 10%, (b) 20%, (c) 25%, (d) 30% and (e) 40% epoxy.

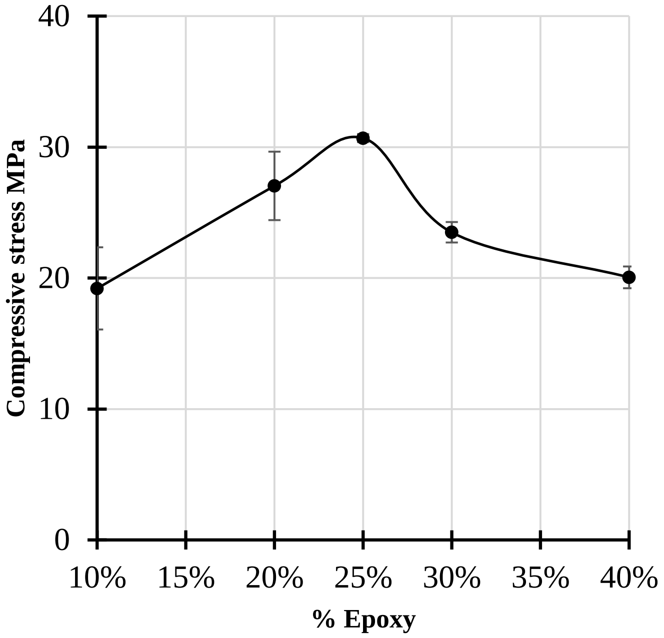

Figure 4.4 Average compressive strength of PC for different percentages of epoxy.

Table 4.2 Compressive strength values for different percentages of epoxy.

Specimen name	Percentage of epoxy	Specimen no	Compressive strength (MPa)	Average compressive strength (MPa)	St. deviation (MPa)
PC1	10%	1	19.06	19.21	3.14
		2	16.14		
		3	22.42		
PC2	20%	1	29.49	27.04	2.61
		2	24.29		
		3	27.35		
PC3	25%	1	30.83	30.70	0.31
		2	30.34		
		3	30.91		
PC4	30%	1	24.29	23.49	0.78
		2	23.45		
		3	22.73		
PC5	40%	1	20.51	20.06	0.83
		2	19.10		
		3	20.56		

Table 4.3 Apparent modulus of elasticity values for different percentages of epoxy.

Specimen name	Percentage of epoxy	Specimen no	AMOE (GPa)	Average AMOE (GPa)	St. deviation (GPa)
PC1	10%	1	1.67	1.61	0.11
		2	1.68		
		3	1.48		
PC2	20%	1	1.51	1.17	0.36
		2	0.80		
		3	1.19		
PC3	25%	1	1.60	1.25	0.36
		2	0.90		
		3	1.14		
PC4	30%	1	0.71	0.81	0.10
		2	0.91		
		3	0.81		
PC5	40%	1	0.52	0.52	0.02
		2	0.50		
		3	0.53		

4.3.1.2 Splitting tensile strength

The results of splitting tensile strength tests are shown in Fig. 4.5. The splitting tensile strength's average values were calculated as 4.84, 8.83, 11.25, 7.73, and 9.01 MPa for 10, 20, 25, 30, and 40% of epoxy content, respectively. The highest average value of splitting tensile strength was 11.25 MPa when 25% of epoxy was used which is the optimum proportion of epoxy in PC for compression test as well. Fig. 4.6 shows a comparison of average splitting tensile strength values for different percentages of epoxy in PC. The maximum values of splitting tensile strengths are used for this graph and are listed in Table 4.4. The average splitting tensile strength versus average compressive strength values of PC for different percentages of epoxy is given in Fig. 4.7. This graph suggests that there is a direct correlation between both compressive and splitting tensile strength values, with the exception of PC5. The correlation coefficient R^2 which is 0.9759 was considered as exceptionally. PC5 with 40% epoxy deviates from a decreasing trend in splitting tensile test. This deviation is considered as an effect of high amount of epoxy in the composition. That is why PC5 is accepted as an outlier in the correlation study.

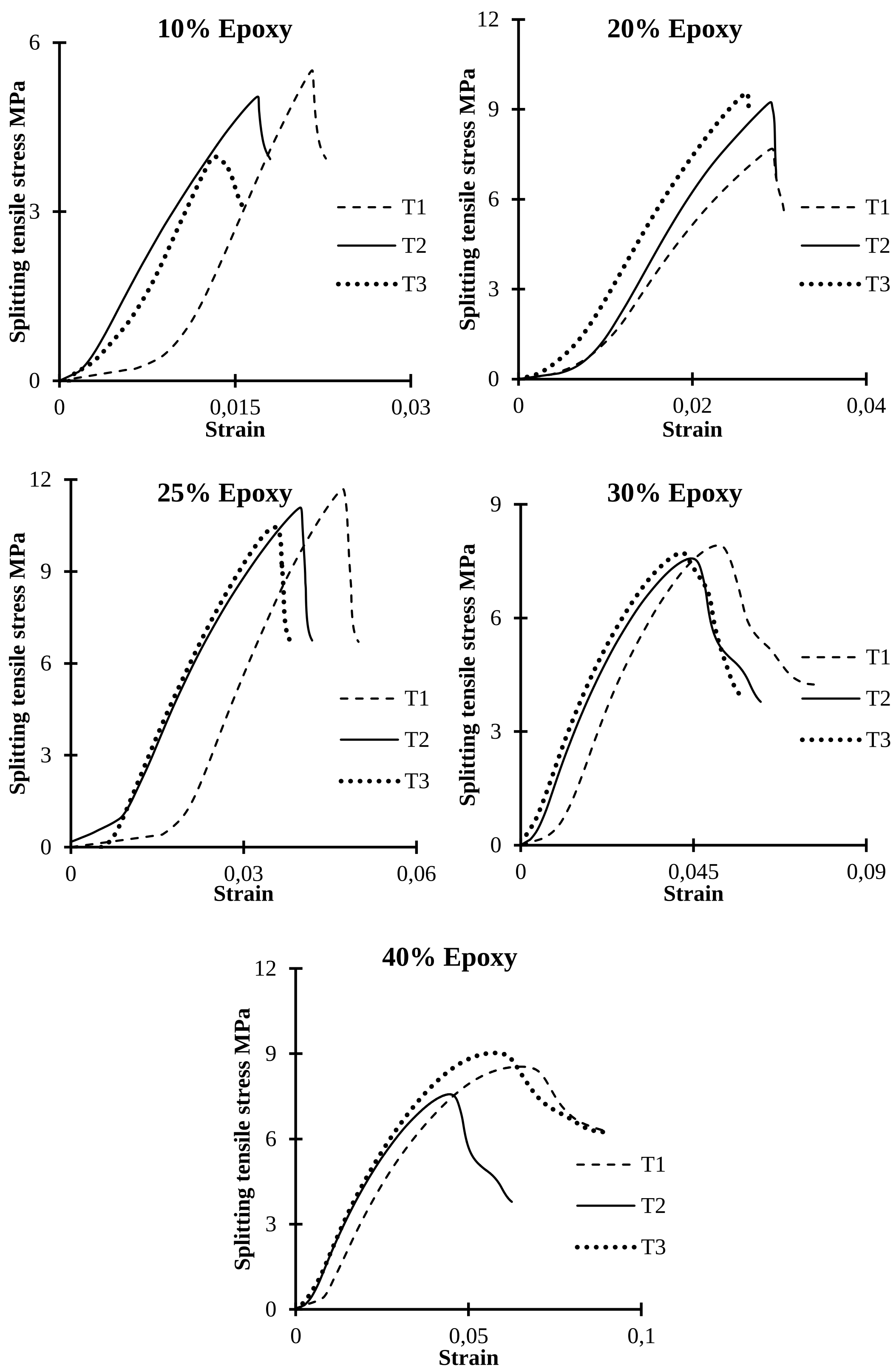

Figure 4.5 Splitting tensile strength test results for (a) 10%, (b) 20%, (c) 25%, (d) 30% and (e) 40% epoxy.

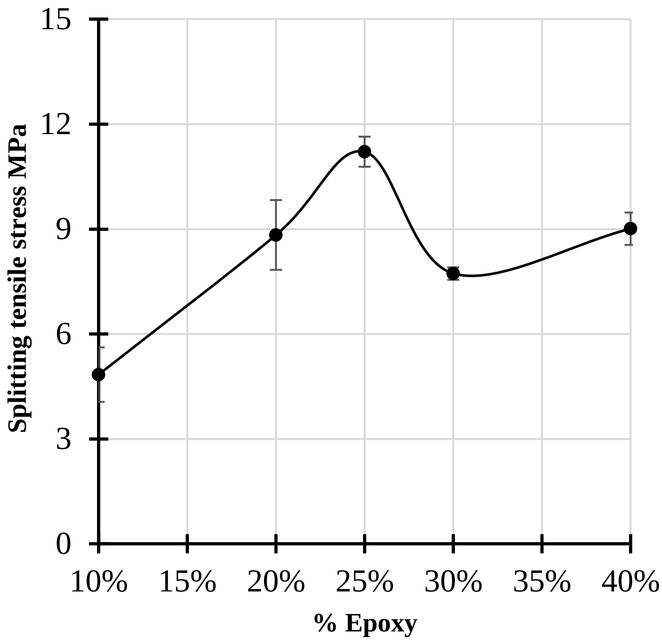

Figure 4.6 Average splitting tensile strength of PC for different percentages of epoxy.

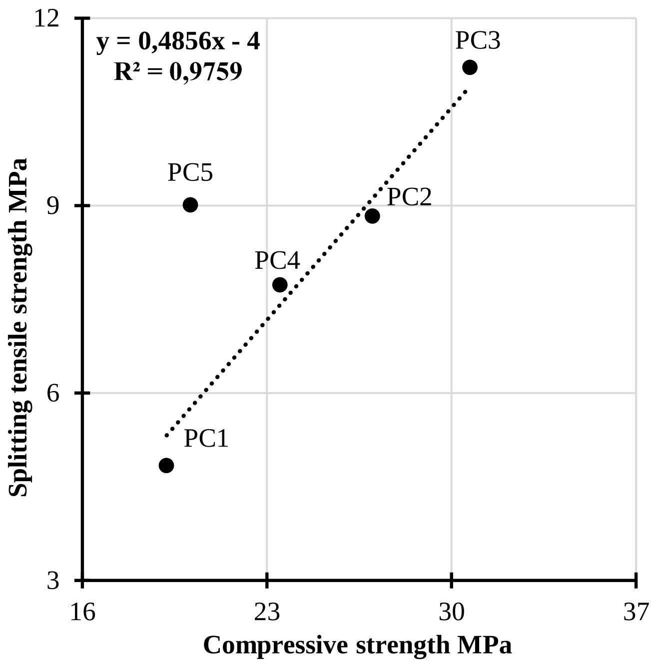

Figure 4.7 Splitting tensile strength versus compressive strength of PC for different percentages of epoxy.

Table 4.4 Splitting tensile strength values for different percentages of epoxy.

Specimen name	Percentage of epoxy	Specimen no	Splitting tensile strength (MPa)	Average splitting tensile strength (MPa)	St. deviation (MPa)
PC1	10%	1	5.49	4.84	0.78
		2	5.04		
		3	3.98		
PC2	20%	1	7.69	8.83	1.00
		2	9.24		
		3	9.55		
PC3	25%	1	11.69	11.21	0.43
		2	11.08		
		3	10.87		
PC4	30%	1	7.92	7.73	0.18
		2	7.57		
		3	7.72		
PC5	40%	1	8.54	9.01	0.46
		2	9.46		
		3	9.03		

4.3.1.3 Flexural strength

Fig. 4.8 shows the results of flexural strength tests of PC samples for given percentages of epoxy. Three different prismatic beams were tested for each composition and the mean values were calculated. As seen from these graphs the flexural strength values were varied between 4.75 and 17.75 MPa depending on the epoxy ratio. The average flexural strength values for 10, 20, 25, 30, and 40% by weight epoxy were calculated as 5.58, 16.10, 15.96, 13.08, and 12.95 MPa, respectively. Notably, the flexural strength values on both 20% and 25% of epoxy were almost the same with a slight preference for 20%. However, since the maximum compressive and splitting tensile strengths were occurred with 25% of epoxy, it is reasonable to choose this composition as the optimum percentage of epoxy for RHA study. The average flexural strength values of PC samples for given percentages of epoxy is shown in Fig. 4.9, and the calculated mean flexural strength values are listed in Table 4.5. The average flexural strength versus average compressive strength values of PC for different percentages of epoxy is given ig Fig. 4.10. Due to the debonding of the aggregates from the epoxy caused by using small amount of epoxy, the specimen with 10% epoxy separated from the trendline hence taken as an outlier. Therefore, when 4 points are considered, a reasonable correlation can be obtained between the compressive strength and flexural strength of the specimens as seen from the graph. The correlation coefficient (R^2) between compressive and splitting tensile strength is 0.793.

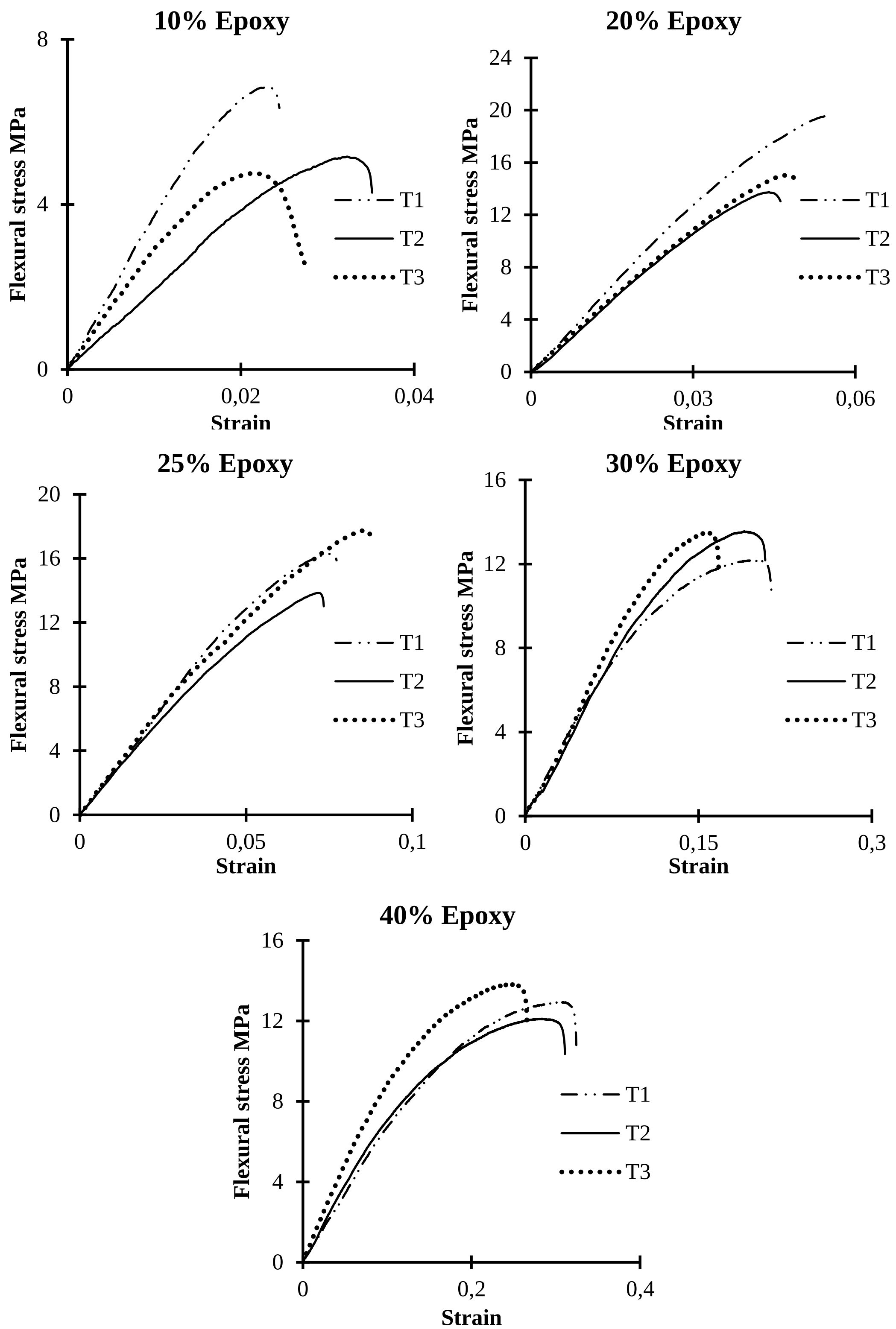

Figure 4.8 Flexural strength test results for (a) 10%, (b) 20%, (c) 25%, (d) 30% and (e) 40% epoxy.

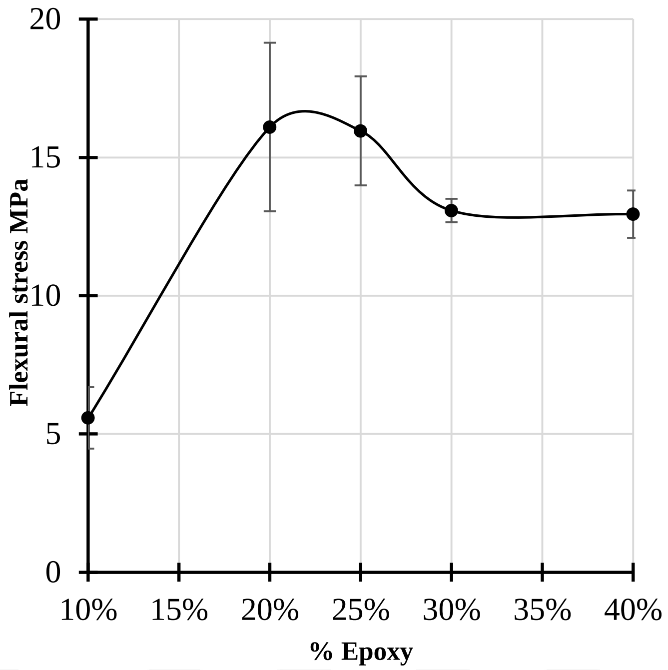

Figure 4.9 Average flexural strength of PC for different percentages of epoxy.

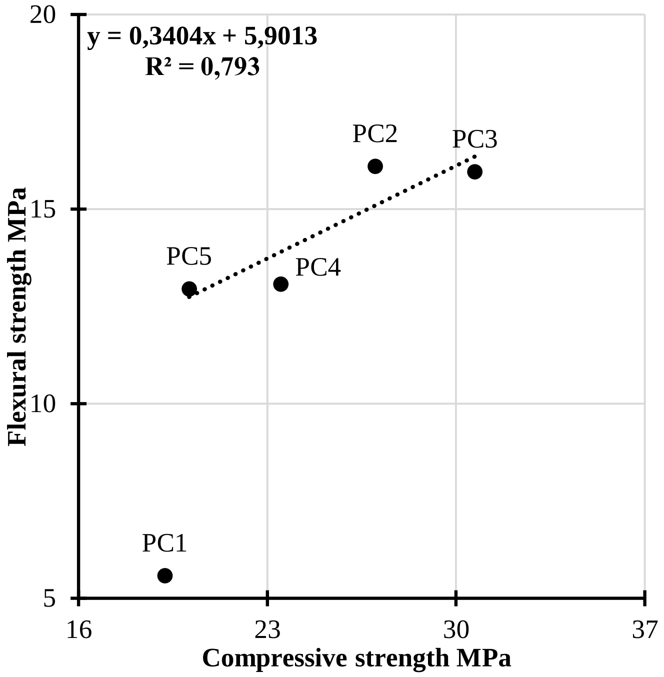

Figure 4.10 Flexural strength versus compressive strength of PC for different percentages of epoxy.

Table 4.5 Flexural strength values for different percentages of epoxy.

Specimen name	Percentage of epoxy	Specimen no	Flexural strength (MPa)	Average flexural strength (MPa)	St. deviation (MPa)
PC1	10%	1	6.85	5.58	1.11
		2	5.16		
		3	4.75		
PC2	20%	1	19.54	16.10	3.05
		2	13.73		
		3	15.04		
PC3	25%	1	16.27	15.96	1.97
		2	13.85		
		3	17.75		
PC4	30%	1	12.81	13.08	0.42
		2	13.55		
		3	13.51		
PC5	40%	1	12.93	12.95	0.86
		2	12.11		
		3	13.82		

4.3.2 The effect of RHA on PC with 3:1 WFS/epoxy ratio

4.3.2.1 Compressive strength

The results of compressive strength tests of each composition set are shown in Fig. 4.11. The percentages of RHA used for optimization study were 5, 10, and 15% of the specimen by weight. Additionally, the compressive strength average values for PC samples with 5, 10, and 15% of RHA were calculated by using maximum values of each sample and determined as 44.34, 51.88, and 37.84 MPa, respectively. It was observed that even as little as 5% addition of RHA into PC mixture enhances the compressive strength compared to PC without RHA, yet, 10% RHA addition made the highest contribution to the compressive strength. Mean compressive strength values for different percentages of RHA is shown in Fig. 4.12, and the mean values of compressive strength are tabulated in Table 4.6. Table 4.7 lists the values of the elasticity modulus of PC samples with 3:1 WFS/epoxy with changing RHA. It was also seen that AMOE reached its maximum value for 10% RHA addition. AMOE is a good indicator of the state of adhesion forces between the components of the mixture.

Thus, it can be concluded that RHA enhances the adhesion forces compared to PC composition without RHA and elevated it to its maximum for 10% addition.

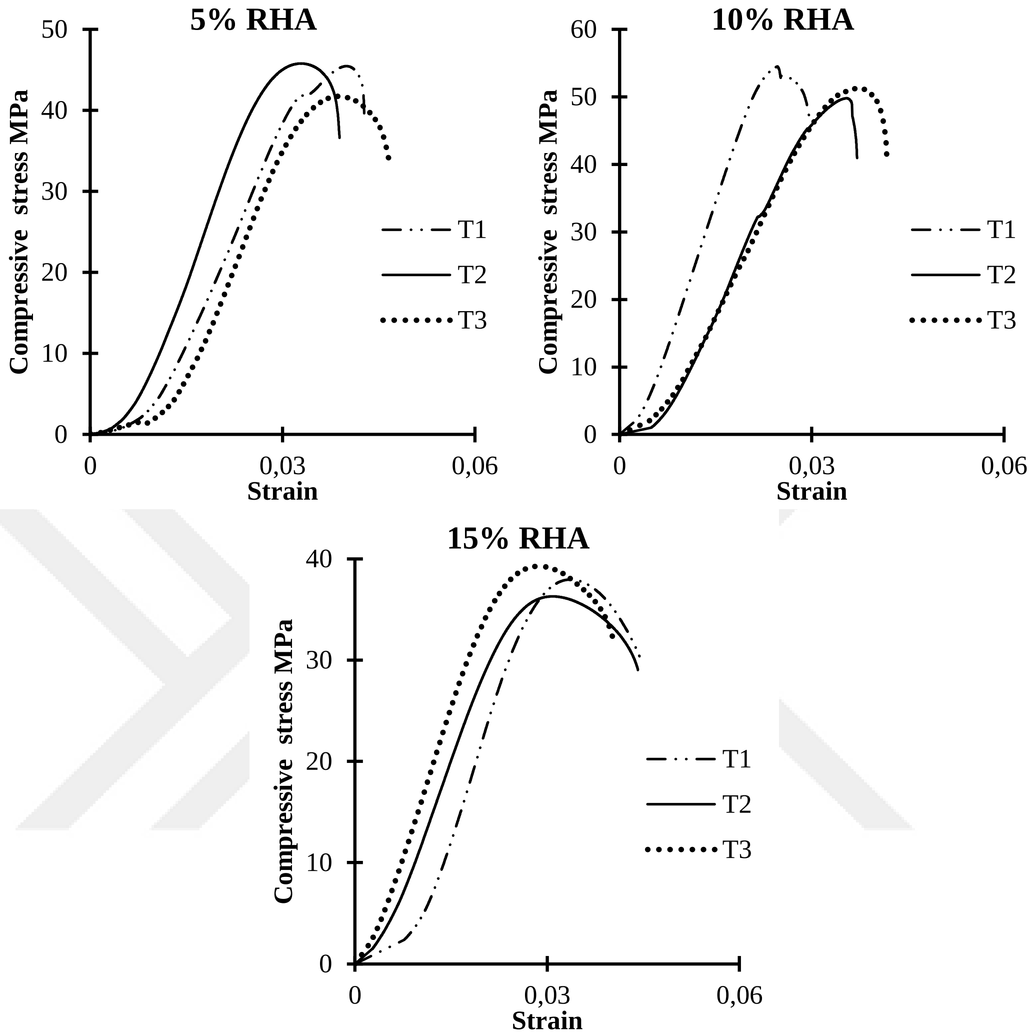

Figure 4.11 Compressive strength test results of PC with 3:1 WFS/epoxy with (a) 5%, (b) 10% and (c) 15% RHA addition.

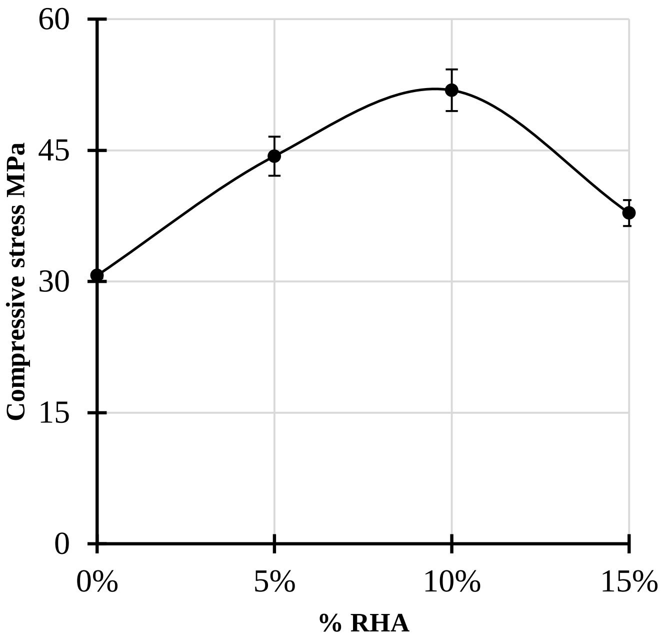

Figure 4.12 Mean compressive strength values of PC for different percentages of RHA.

Table 4.6 Compressive strength values of PC with various RHA addition.

Specimen name	Percentage of RHA	Specimen no	Compressive strength (MPa)	Average compressive strength (MPa)	St. deviation (MPa)
PC3	0%	1	30.83	30.70	0.31
		2	30.34		
		3	30.91		
PC6	5%	1	45.47	44.34	2.24
		2	45.80		
		3	41.76		
PC7	10%	1	54.54	51.88	2.37
		2	49.81		
		3	51.88		
PC8	15%	1	37.96	37.84	1.49
		2	36.29		
		3	39.26		

Table 4.7 Apparent modulus of elasticity of PC with various RHA.

Specimen name	Percentage of RHA	Specimen no	AMOE (GPa)	Average AMOE (GPa)	St. deviation (GPa)
PC3	0%	1	1.60	1.21	0.36
		2	0.90		
		3	1.14		
PC6	5%	1	1.81	1.84	0.12
		2	1.98		
		3	1.74		
PC7	10%	1	2.78	2.16	0.54
		2	1.91		
		3	1.8		
PC8	15%	1	1.81	1.81	0.09
		2	1.89		
		3	1.72		

4.3.2.2 Splitting tensile strength

The splitting tensile strength values for different RHA content are shown in Fig. 4.13. The results show that the increase of RHA to 10% has made the highest contribution to the splitting tensile strength. The values of splitting tensile strength test results were varied between 10.00 and 13.00 MPa approximately. It can be concluded that the addition of 5% and 10% of RHA made almost the same contribution to splitting tensile strength with 12.31 and 12.51 MPa, respectively and these values are slightly higher than PC without RHA. Further contribution of RHA caused a slight decrease from the maximum value. It should be noted that all these changes are so slight within the range of standard deviation values. So, it can be concluded that the RHA effect on splitting tensile strength is not as statistically significant as it was for compressive strength. Average splitting tensile strength values for different percentages of RHA is given in Fig. 4.14, and the mean splitting tensile strength values calculated from the maximum values of each sample are tabulated in Table 4.8.

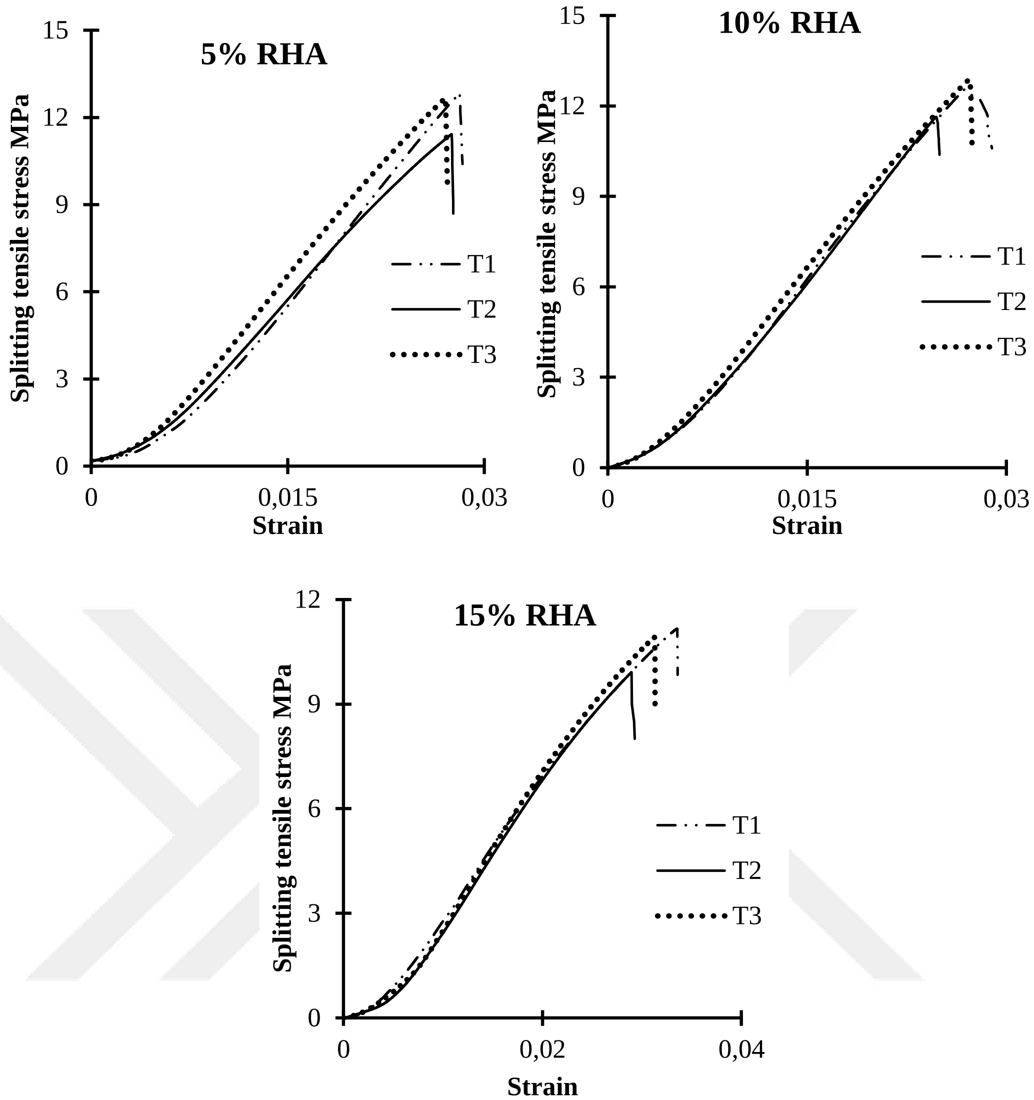

Figure 4.13 Splitting tensile strength test results of PC with 3:1 ratio of WFS/epoxy with (a) 5%, (b) 10% and (c) 15% RHA addition.

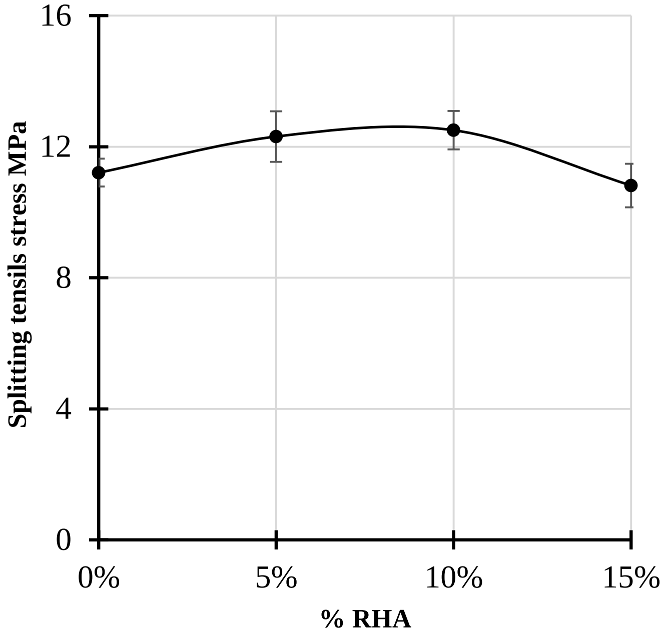

Figure 4.14 Average splitting tensile strength values for different percentages of RHA.

Table 4.8 Splitting tensile strength values of PC with various RHA.

Specimen name	Percentage of RHA	Specimen no	Splitting tensile strength (MPa)	Average splitting tensile strength (MPa)	St. deviation (MPa)
PC3	0%	1	11.69	11.21	0.43
		2	11.08		
		3	10.87		
PC6	5%	1	12.84	12.31	0.78
		2	11.42		
		3	12.67		
PC7	10%	1	12.72	12.51	0.59
		2	11.84		
		3	12.96		
PC8	15%	1	11.32	10.82	0.67
		2	10.06		
		3	11.07		

4.3.2.3 Flexural strength

Fig. 4.16 shows the flexural strength values of PC with 3:1 WFS/epoxy ratio with respect to various RHA values. The flexural strength values are varied between 8.00 and 22.50 MPa approximately. The average flexural strength values of PC are 21.31, 21.74 and 9.44 MPa for 5, 10, and 15% of RHA addition, respectively. Maximum increase in flexural strength value was observed for 10% with RHA. However, there is not much statistically significant difference between 5% and 10% RHA addition when standard deviation amounts were taken into account. This behavior is quite similar to the one observed for splitting tensile strength test. Yet the increase in flexural strength with RHA addition compared to PC without RHA is more pronounced than splitting tensile test. Comparing different percentages of RHA in PC for flexural strength is shown in Fig. 4.17, and the mean values of flexural strength tests are listed in Table 4.9. In addition to these, the average density of the optimum mix is found around 1.6g/cm^3.

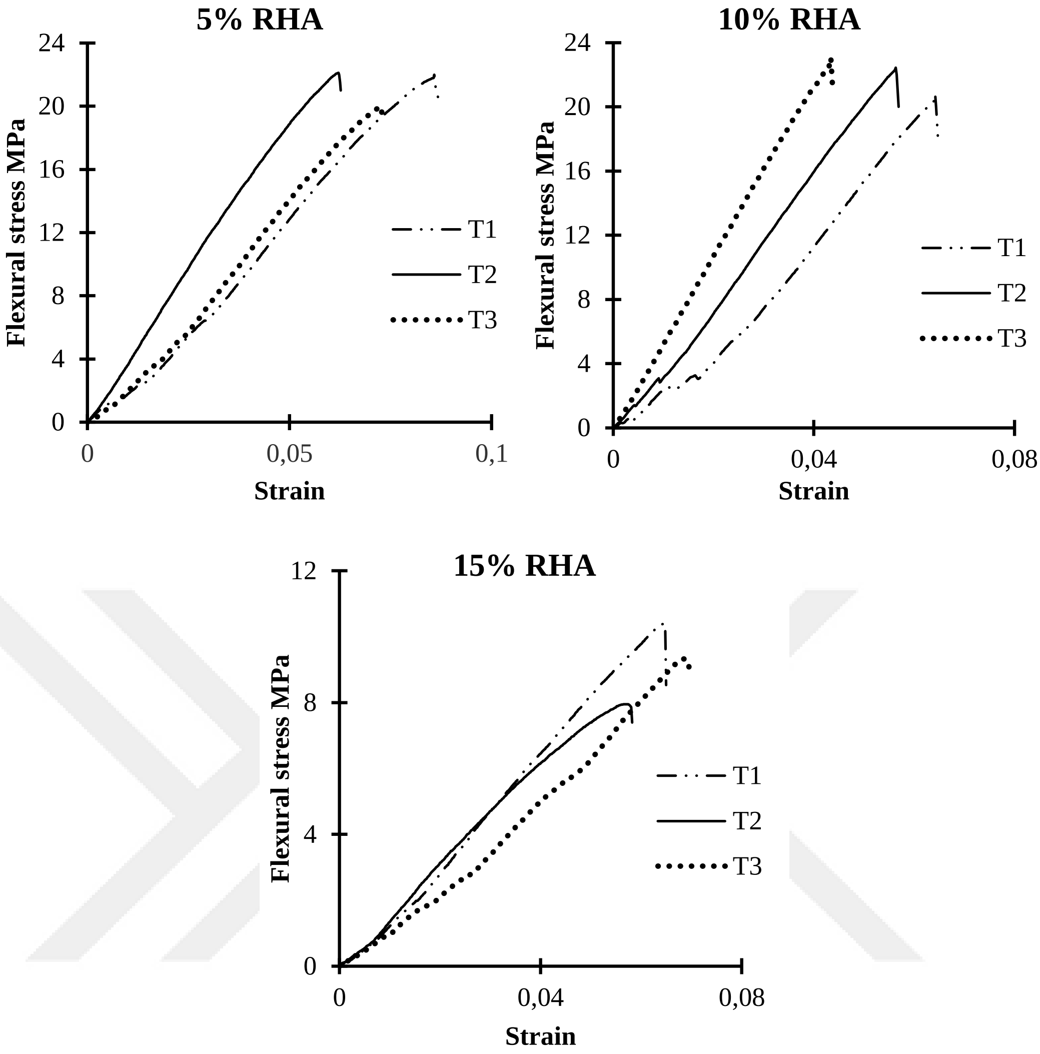

Figure 4.15 Flexural strength test results of PC with 3:1 WFS/epoxy ratio and (a) 5%, (b) 10% and (c) 15% RHA addition.

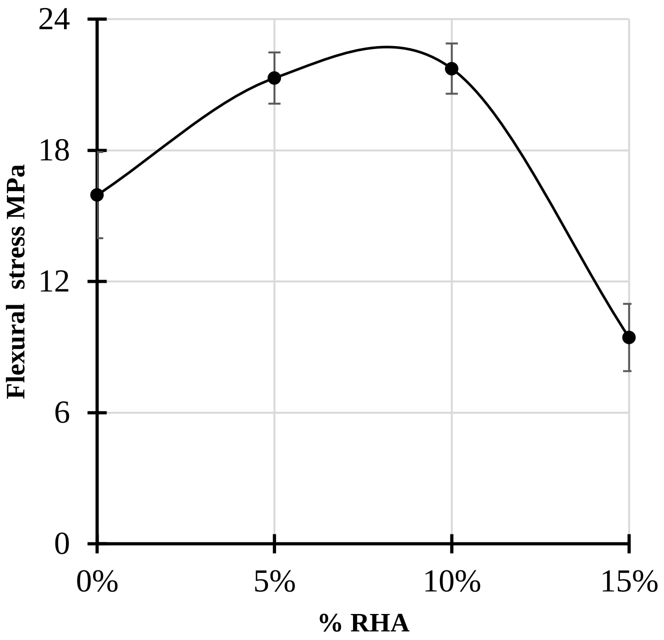

Figure 4.16 Average flexural strength values for different percentages of RHA.

Table 4.9 Flexural strength values of PC with various RHA.

Specimen name	Percentage of RHA	Specimen no	Flexural strength (MPa)	Average flexural strength (MPa)	St. deviation (MPa)
PC3	0%	1	16.27	15.96	1.97
		2	13.85		
		3	17.75		
PC6	5%	1	21.85	21.31	1.18
		2	22.11		
		3	19.95		
PC7	10%	1	20.41	21.74	1.15
		2	22.40		
		3	22.42		
PC8	15%	1	11.03	9.44	1.54
		2	7.95		
		3	9.33		

4.3.3 The effect of RHA on PC with 3:1 NS/epoxy ratio

4.3.3.1 Compressive strength

Compressive strength test results are shown in Fig. 4.19. One of the sets was prepared without RHA using the optimum percentage of epoxy which is 25%. The rest of the sets were prepared with RHA additions of 5, 10, and 15% by weight. The values were varied between 13.0 and 47.5 MPa, approximately. It was observed that WFS improves the compressive strength of PC better than NS by around 4 MPa. It is thought that this 4 MPa difference between two sands happens due to the carbonized resin compounds enveloping the sand grains in WFS. This naturally surrounding hydrocarbon layer around WFS grains behaves like some kind of compatibilizer and thus establishes a stronger adhesion between the WFS and epoxy resin which itself a polymeric hydrocarbon. Carbon residues behave as compatibilizers which decrease surface energy of the sand grains by allowing wetting of the sand grains by epoxy resin and hence increase the adhesion forces. This phenomenon also provides better homogeneity of the PC mixture based on the “like dissolves like” principle. Another thing that also supported this idea is the fact WFS has less water content compared to NS. Likewise, compressive strength values of NS added PC improves up to 10% RHA addition, as it did before with WFS added PC. Average compressive strength values for different percentages of RHA are shown in Fig. 4.20. Compressive strength average values for WFS and NS are compared in Fig. 4.21 and the mean values of compressive strengths calculated from maximum values are tabulated in Table 4.10. In addition, Table 4.11 lists the values of AMOE of NS added PC samples with changing RHA ratios. AMOE of PC with 10% RHA increased from 1.88 GPa to 2.16 GPa when NS is replaced by WFS, which also indicates a significant improvement in adhesion forces between composite components of the PC matrix when WFS is used.

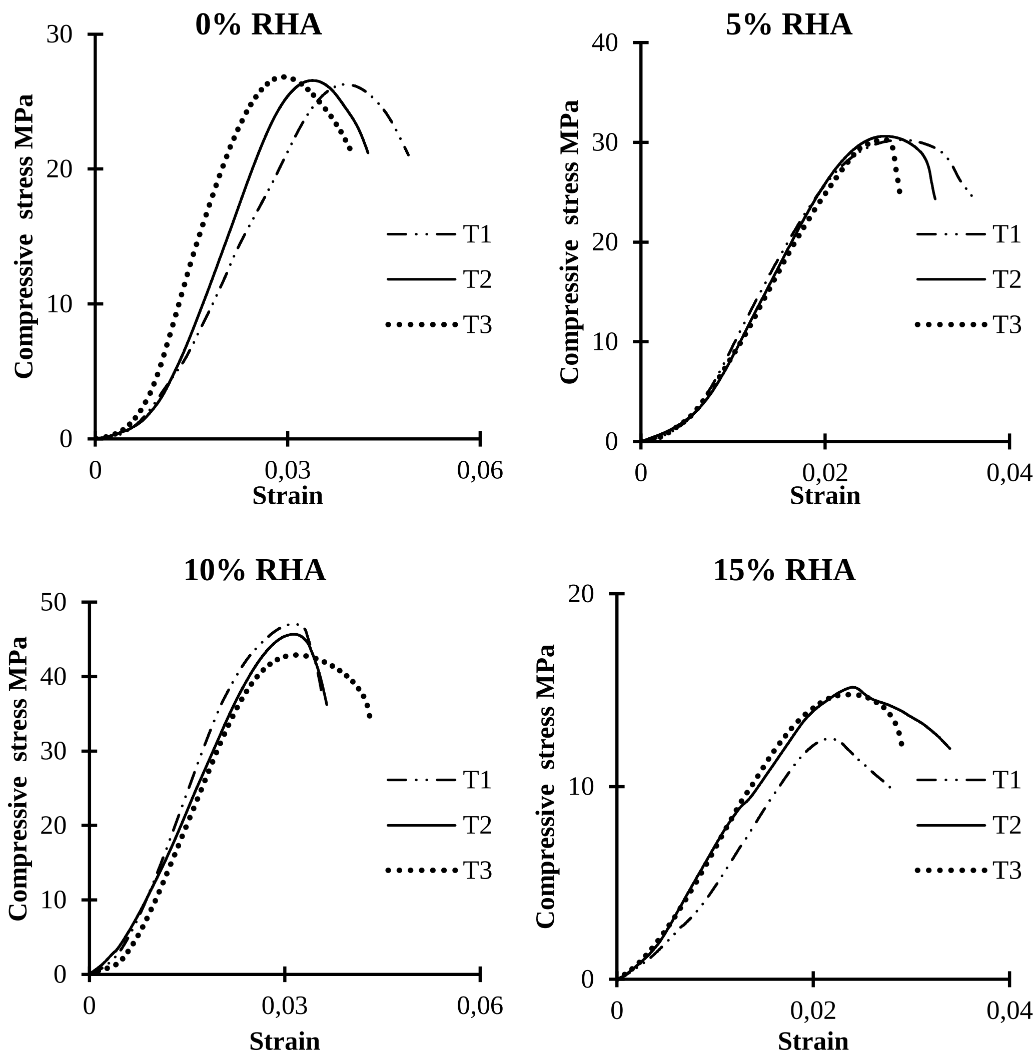

Figure 4.17 Compressive strength test results of PC with 3:1 NS/epoxy ratio with (a) 0%, (b) 5%, (c) 10% and (d) 15% RHA addition.

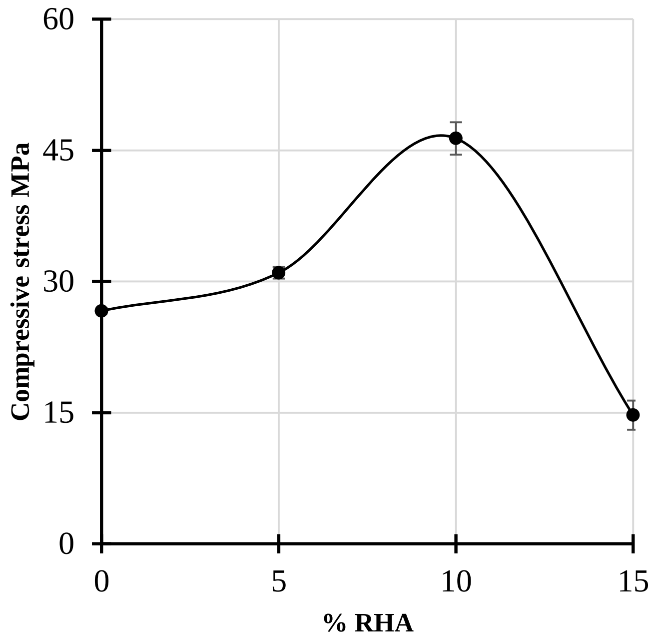

Figure 4.18 Average compressive strength values of PC with 3:1 NS/epoxy ratio for different percentages of RHA.

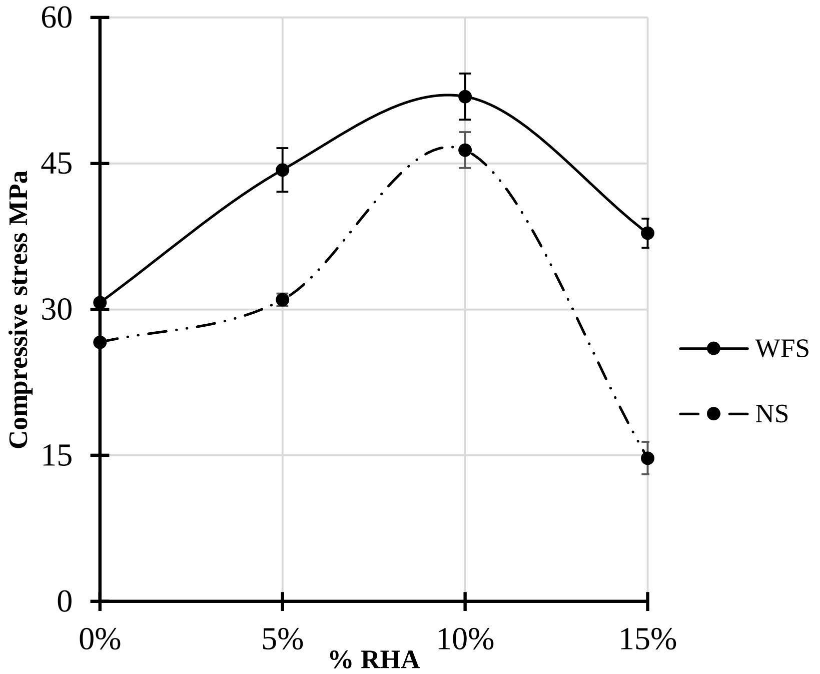

Figure 4.19 Mean compressive strength values for WFS and NS added PC.

Table 4.10 Compressive strength values of PC with 3:1 NS/epoxy ratio and various RHA ratio.

Specimen name	Percentage of RHA	Specimen no	Compressive strength (MPa)	Average compressive strength (MPa)	St. deviation (MPa)
PC9	0%	1	26.28	26.64	0.31
		2	26.82		
		3	26.83		
PC10	5%	1	31.21	30.97	0.65
		2	31.52		
		3	30.28		
PC11	10%	1	47.45	46.38	1.85
		2	47.43		
		3	44.24		
PC12	15%	1	12.82	14.37	1.67
		2	15.88		
		3	15.49		

Table 4.11 Apparent modulus of elasticity of PC with 3:1 NS/epoxy ratio with various RHA.

Specimen name	Percentage of RHA	Specimen no	Apparent AMOE (GPa)	Average AMOE (GPa)	St. deviation (GPa)
PC9	0%	1	0.82	0.82	0.08
		2	0.73		
		3	0.89		
PC10	5%	1	1.56	1.60	0.08
		2	1.69		
		3	1.56		
PC11	10%	1	1.95	1.88	0.07
		2	1.81		
		3	1.88		
PC12	15%	1	0.79	0.77	0.09
		2	0.67		
		3	0.85		

4.3.3.2 Splitting tensile strength

The splitting tensile strength test results are shown in Fig. 4.22. The first set was prepared without RHA using the optimum percentage of epoxy which is 25%. The splitting tensile strength values were varied between 3.5 and 13.5 MPa approximately, as a function of the RHA ratio. Splitting tensile strength values increased with the increase of RHA up to 10%. Furthermore, the average results are 8.29, 9.52, 12.56 and 5.13 MPa for 0, 5, 10 and 15% of RHA, respectively. The maximum value of splitting tensile strength was observed at 10% of RHA addition for both NS added and WFS added PC. However, the effect of RHA on splitting tensile strength for NS added PC is more dramatic compared to a slight change in WFS added PC. Another interesting phenomenon was observed that is the similarity of the maximum mean values of splitting tensile strength for NS added PC and WFS added PC with 10% RHA addition. They are almost same. Average splitting tensile strength values for different percentages of RHA are shown in Fig. 4.23. The difference between splitting tensile strength average values for WFS and NS added PCs is shown in Fig. 4.24. The mean values of splitting tensile strengths are listed in Table 4.12.

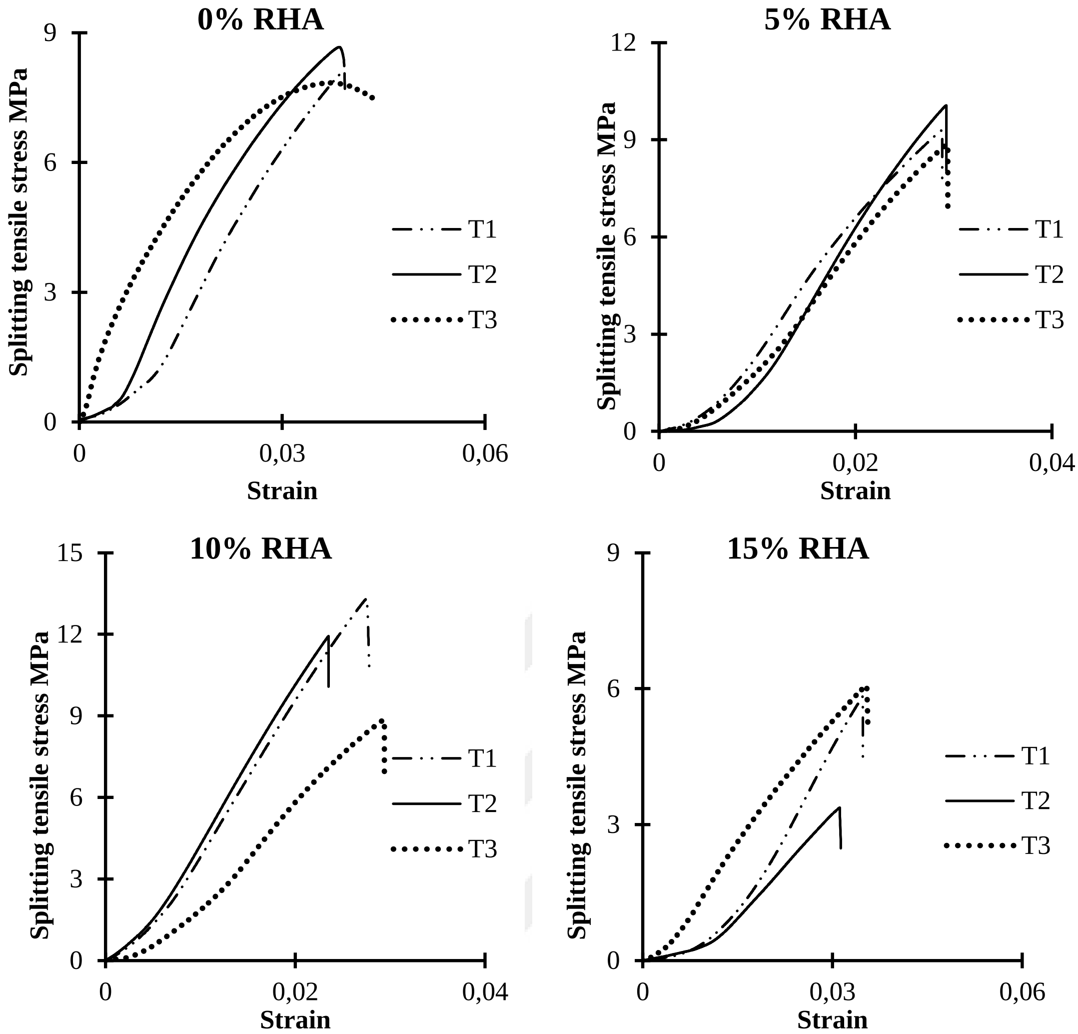

Figure 4.20 Splitting tensile strength results of PC with 25% epoxy and 3:1 NS/epoxy ratio with (a) 0%, (b) 5%, (c) 10% and (d) 15% RHA addition.

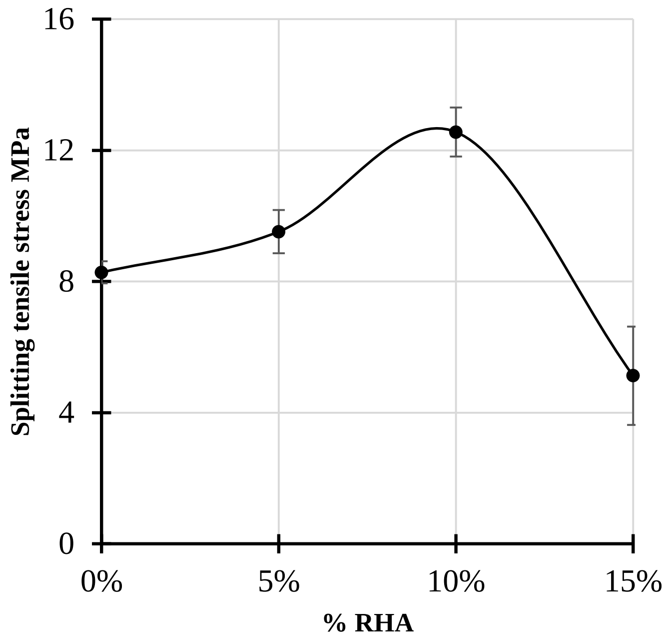

Figure 4.21 Effect of different percentages of RHA in NS added PC on splitting tensile strength values.

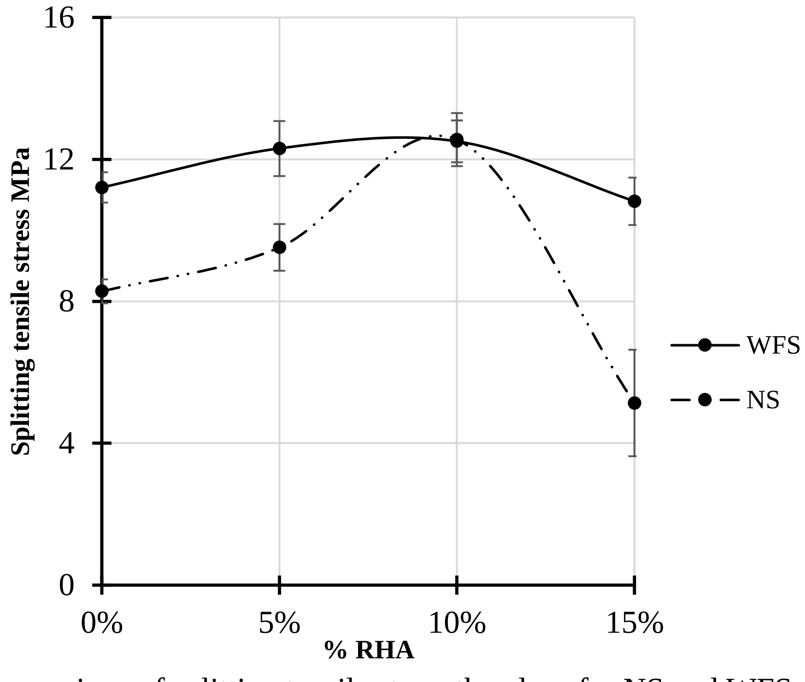

Figure 4.22 Comparison of splitting tensile strength values for NS and WFS added PCs with 3:1 sand/epoxy ratio and various RHA additions.

Table 4.12 Splitting tensile strength values of NS added PC with RHA.

Specimen name	Percentage of RHA	Specimen no	Splitting tensile strength (MPa)	Average splitting tensile strength (MPa)	St. deviation (MPa)
PC9	0%	1	8.12	8.29	0.34
		2	8.67		
		3	8.05		
PC10	5%	1	9.41	9.52	0.66
		2	10.22		
		3	8.92		
PC10	10%	1	13.42	12.56	0.75
		2	12.22		
		3	12.05		
PC11	15%	1	5.87	5.13	1.50
		2	3.41		
		3	6.13		

4.3.3.3 Flexural strength

Fig. 4.25 shows the flexural strength values of PC with 3:1 NS/epoxy ratio with various RHA. The first set was prepared without RHA. As can be seen, the flexural strength values are varied between 3.5 and 20.0 MPa approximately as a function of RHA amount. 15.47, 17.35, 19.42, and 5.93 MPa were the mean flexural strength values corresponding to 0, 5, 10, and 15% of RHA, respectively. Effect of different percentages of RHA in NS added PC on flexural strength is shown in Fig. 4.26. and then it is compared with WFS added PC in Fig. 4.27. The values of flexural strength tests are listed in Table 4.13.

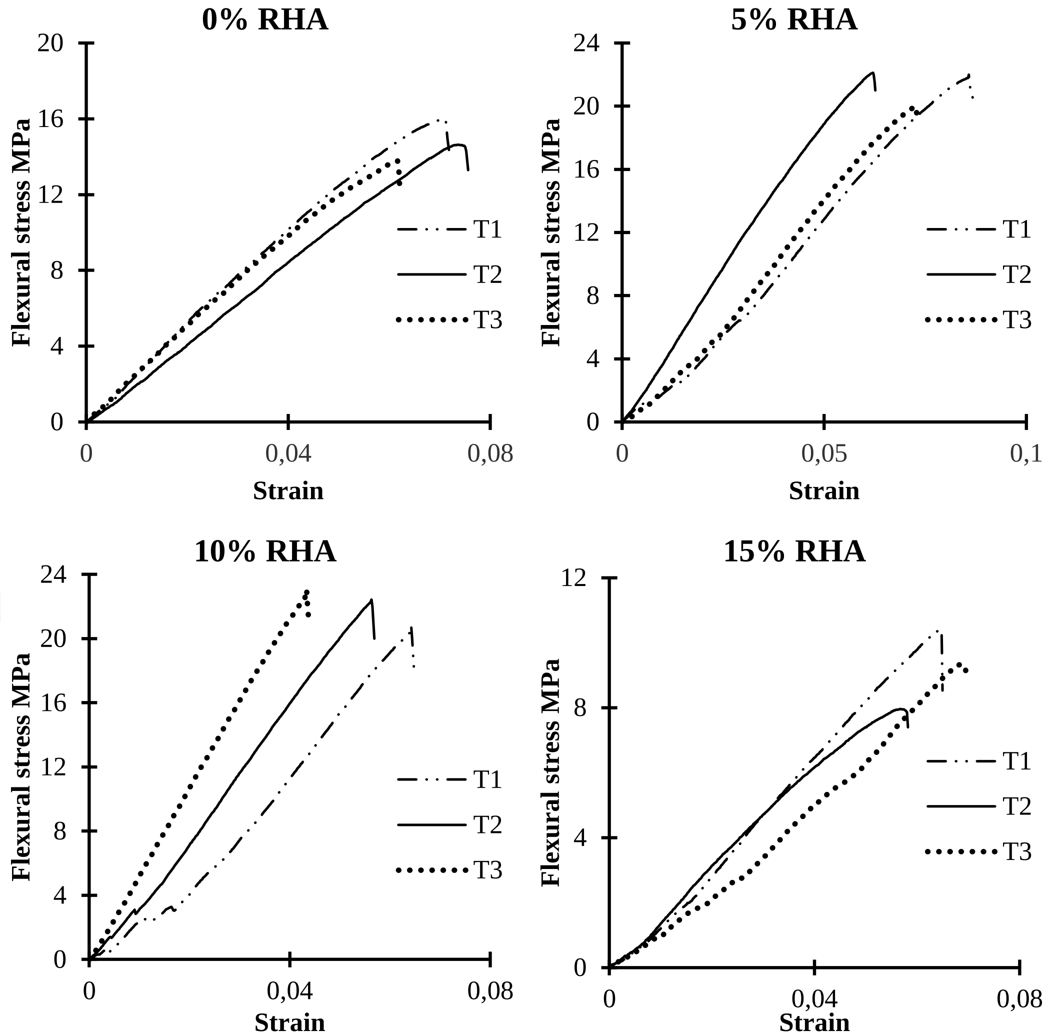

Figure 4.23 Flexural strength test results of PC with 25% epoxy and NS with (a) 0%, (b) 5%, (c) 10% and (d) 15% RHA.

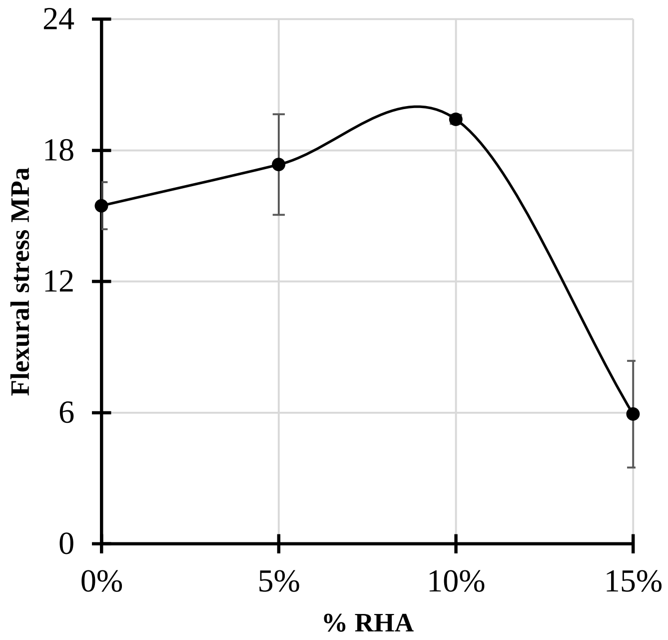

Figure 4.24 Average flexural strength values for different percentages of RHA.

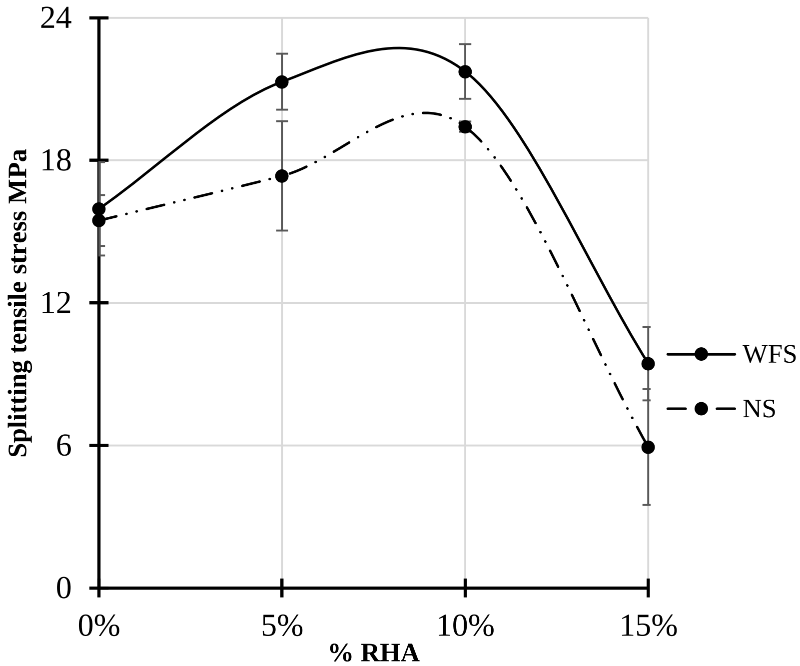

Figure 4.25 Variation of flexural strength values of NS and WFS added PCs.

Table 4.13 Flexural strength values of NS added PC with RHA.

Specimen name	Percentage of RHA	Specimen no	Flexural strength (MPa)	Average flexural strength (MPa)	St. deviation (MPa)
PC9	0%	1	16.61	15.47	1.07
		2	15.33		
		3	14.48		
PC10	5%	1	16.56	17.35	2.30
		2	15.55		
		3	19.95		
PC11	10%	1	19.26	19.42	0.22
		2	19.34		
		3	19.67		
PC12	15%	1	8.31	5.93	2.44
		2	3.43		
		3	6.05		

4.4 X- Ray fluorescence (XRF) spectroscopy

XRF spectroscopy was used to determine the chemical composition of WFS, NS, and RHA. Clearly, WFS is rich in Si compared to NS. However, XRF could not detect the carbon compound residues adsorbed on the sand grains which is probably one of the reasons together with high Si content that WFS shows better compressive strength and modulus of elasticity compared to NS. When it comes to RHA, it can be observed that RHA is a rich in silica – above 87%. Since silica is the main element that increases the concrete strengths, it could be one of the reasons for the observed enhancement of PC which contains WFS (Nair et al. 2008). The chemical composition of WFS, NS, and RHA is shown in Table 4.14.

Table 4.14 Chemical composition of WFS, NS, and RHA.

Element	Weight (%)		
	WFS	NS	RHA
Si	98.27	55.96	87.21
Al	0.44	6.81	0.32
Fe	0.39	7.32	0.57
Ca	0.35	26.98	3.19
P	0.10	0.35	6.13
Ti	0.09	0.73	0.03
Mn	0.04	0.16	0.30
Mg	0.01	1.24	0.66
S	-	0.03	1.21
Cr	0.01	0.25	-

4.5 Optical and scanning electron microscopy (SEM) images of fracture surfaces

The microstructure of the fracture surfaces of PC specimens with 0, 5, 10, and 15% RHA were first observed by optical methods as shown in Fig. 4.28 and then analyzed by SEM. Figures 4.29 – 4.32 show the SEM micrographs of the fractured surfaces of PC under 30, 100 and 200X magnifications. In addition, the scale bars are 1, 4 mm, and 500 µm for 30, 100, and 200X, respectively. Also, it can be observed that samples with a low amount of RHA have larger pore sizes as well as the high number of pores. Fig. 4.29 shows that PC without RHA has a porous network structure throughout its fracture surface which mechanically weakens the PC. Indeed, the interface between the WFS grains and resin is easily detectable under SEM where sand boundaries are sharp and clean, suggesting that WFS is not strongly bonded to epoxy resin in the absence of RHA which affects its mechanical properties negatively. On the other hand, the specimen with 15% of RHA content has very few voids or pores with hardly detectable dimensions under same magnifications. Yet, even though its mechanical properties are better than PC without RHA, it is not as good as 10% RHA added PC. In Fig. 4.30 and Fig. 4.31, as RHA amount increases the pore number and size decreases which is an indication of elevated adhesive forces between the resin and the WFS. Even though it was expected that better adhesion between WFS and epoxy resin with 15% RHA where sand grains cannot be seen individually as shown in Fig. 4.32, the optimum proportion of RHA in PC is 10% RHA with the highest values in all mechanical properties. 10% RHA added PC fracture surface is seen in Fig. 4.31. Therefore, it is thought that there is a golden ratio where mechanical improvement reaches its peak once the adhesive forces between epoxy resin and WFS reached their maximum. If RHA is continuously being added beyond that point, mechanical properties start deteriorating due to shadowing of adhesive forces between resin matrix and WFS reinforcing filler by overused RHA.

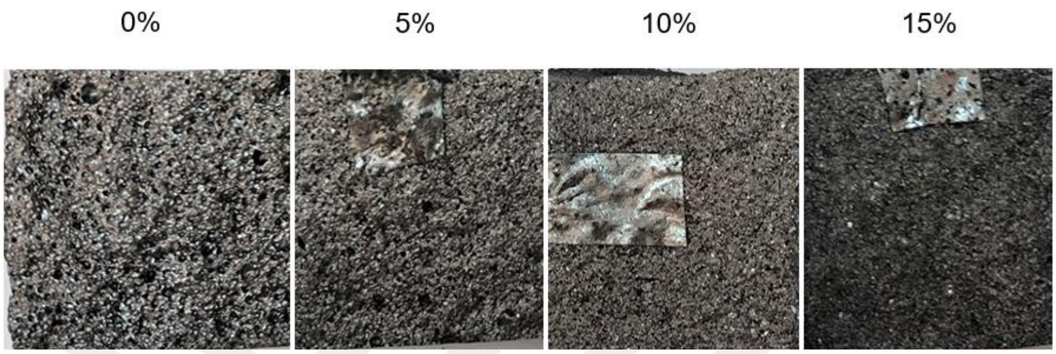

Figure 4.26 Optical images of PC fracture surfaces.

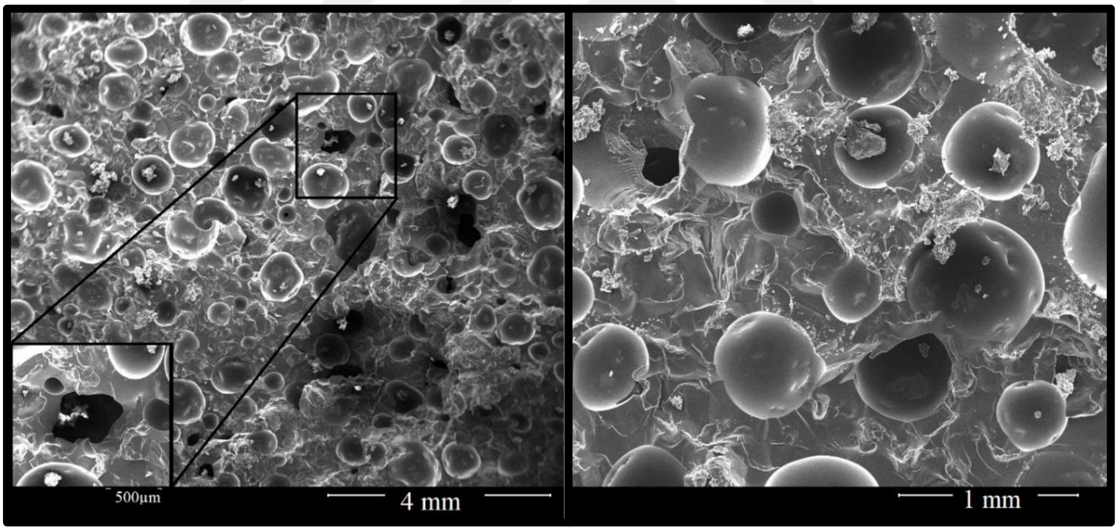

Figure 4.27 SEM micrographs of PC without RHA.

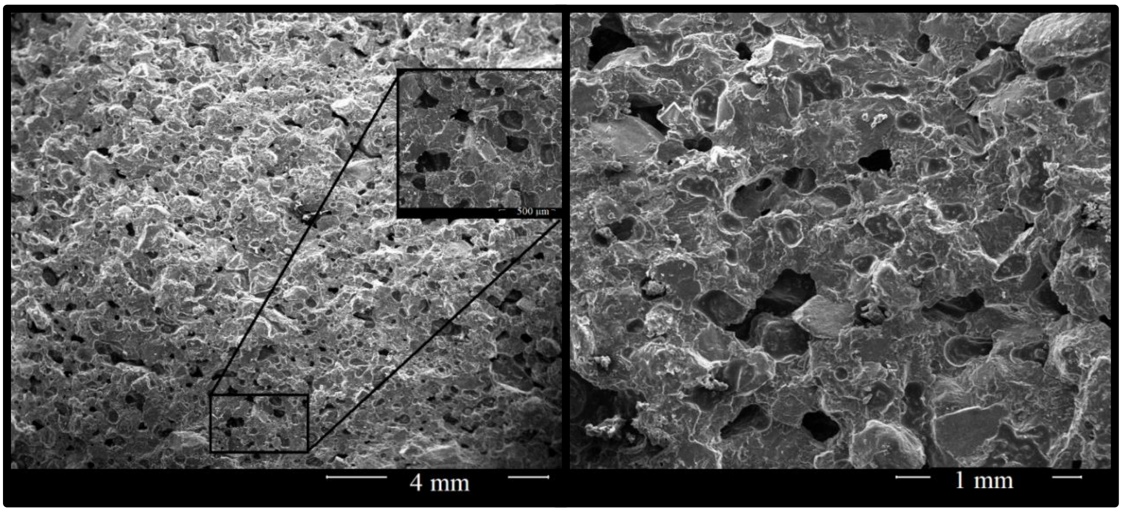

Figure 4.28 SEM micrograph of PC with 5% of RHA.

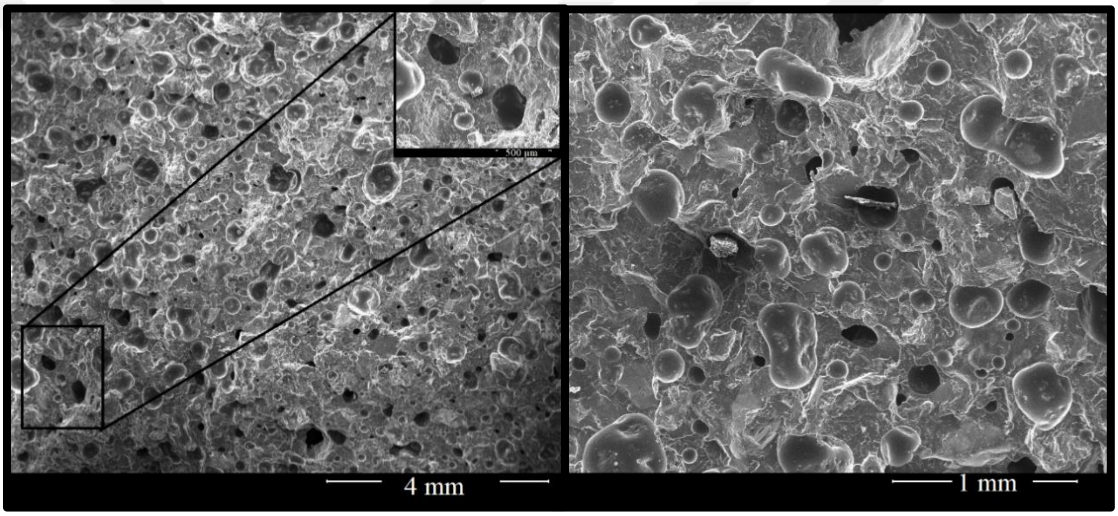

Figure 4.29 SEM micrograph of PC with 10% of RHA.

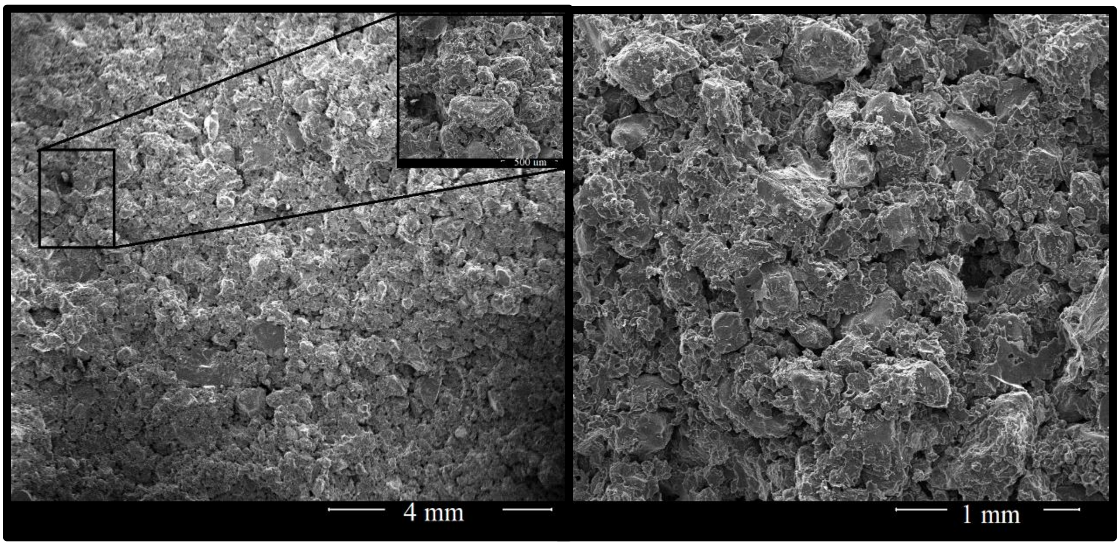

Figure 4.30 SEM micrograph of PC with 15% of RHA.

4.6 Attenuated total reflectance-Fourier transform infrared spectroscopy

In order to determine the infused carbon compounds of WFS and compare it with NS, ATR-FTIR analysis was done. Fig. 4.31 shows FTIR spectrometer plot of both WFS and NS. Fig. 4.32 and Fig. 4.33 show the curve fitting for WFS and NS. It can be detected from Fig. 4.34 there is a peak near 1200 cm^{-1} in WFS compared to NS. This band is a strong indicator of CH_2 wagging (out of plane bending motion) as well stretching motion of C-O bonds like in phenyl ethers or acetate esters. This band is not detected in NS which proves that there are carbon compound residues on WFS grains left over from phenolic resin which is frequently used in sand mold preparation and burnt out during metal casting stage. This argument is also supported with least water absorption of WFS as well. This clearly indicates that WFS is more hydrophobic compared to NS. Therefore, WFS has more tendency for adhesion to epoxy resin which is believed as the reason for its better mechanical properties compared to NS. Table 4.15 shows the characteristic peaks for WFS and NS.

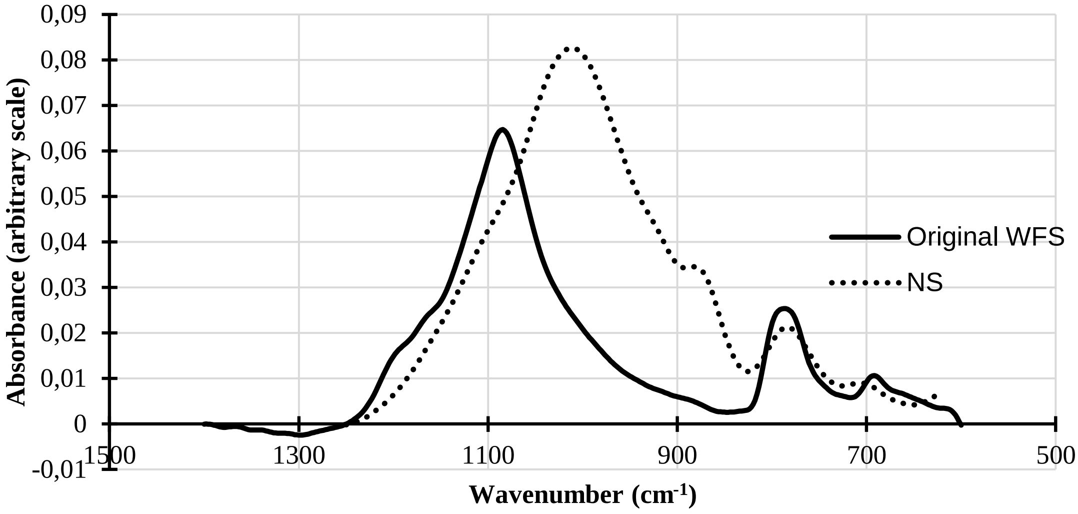

Figure 4.31 ATR-FTIR spectrum of WFS and NS.

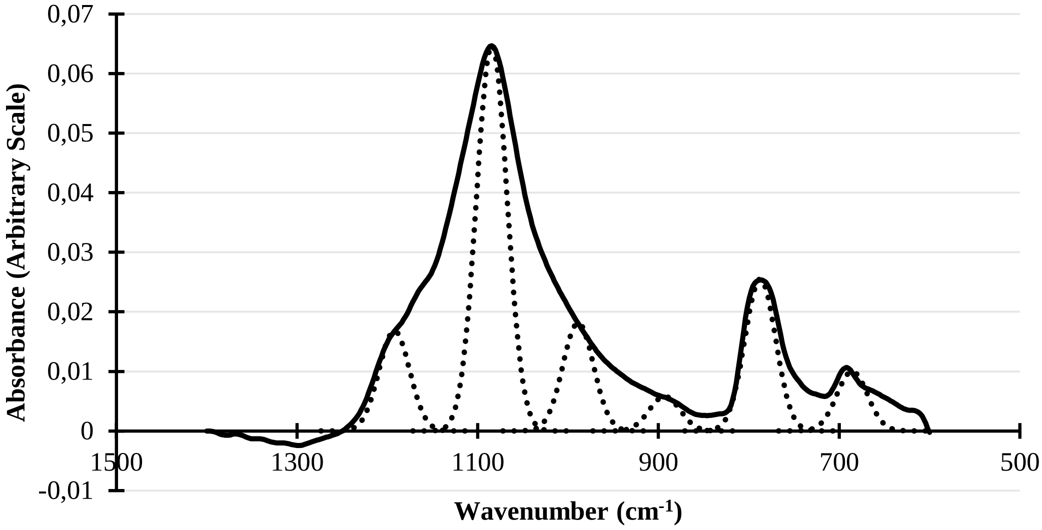

Figure 4.32 Curve fitting of WFS.

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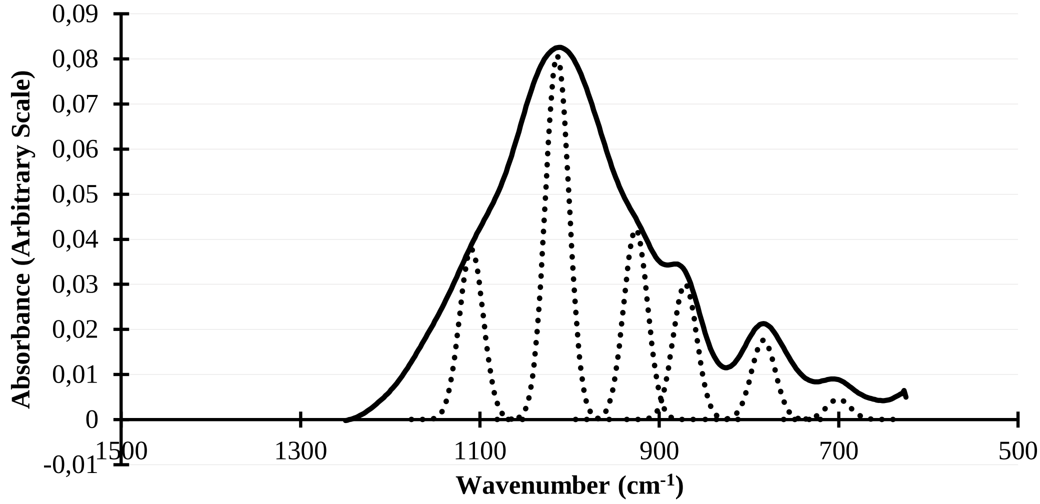

Figure 4.33 Curve fitting of NS.

Table 4.15 Summary of peaks of WFS and NS**.**

FTIR Bands	WFS (cm^{-1})	NS (cm^{-1})
1	685.496	700.324
2	787.277	783.790
3	893.13	871.335
4	988.804	926.303
5	1084.478	1013.844
6	1192.366	1109.528

CHAPTER 5

CONCLUSION

5.1 Conclusion

This investigation was carried out according to various ASTM standards to obtain an optimum PC mixture with high-performance characteristics by using epoxy, WFS, and RHA. The compressive, splitting tensile and flexural strength tests were used in order to determine the optimum percentages of epoxy/WFS and RHA. Also, SEM images were taken from fracture surfaces to investigate the effect of RHA on the microstructure formation and hence on the adhesive bonds between the resin and WFS. Then, XRF was used to determine the elemental compositions together with ATR-FTIR to detect the carbon compound residues around the sand grains.

Based on the presented results it can be concluded that:

- 25% epoxy by weight without RHA showed the best mechanical properties for the PC and thus all PC samples were prepared according to 3:1 WFS/epoxy ratio.
- Addition of 10% RHA by weight as filler to 3:1 WFS/epoxy mixture increased compressive, splitting tensile and flexural strengths by 71, 11, and 35% respectively. Indeed, all these properties reached their peak values at that RHA ratio which was accepted as the optimum amount of the RHA.
- PC which contains WFS showed substantially better compressive strength values than the one which contains NS. On the other hand, there is significant improvement in flexural strength but a slight increase in splitting tensile strength whence WFS is used instead of NS in PC. This quite improvement in mechanical properties with WFS could be due to the burned organic resins which are used in sand casting mold preparation during metal casting and left as carbon compound residues surrounding WFS grains.

- SEM micrographs showed that the PC microstructures were quite homogeneous. Also, the void size and number decrease with increasing RHA ratio.
- PC can be a good alternative as a repair mortar especially when waste materials are used.
- Due to its high bonding capability to cement concrete as well as steel reinforcement with its low density as $1.6 g/cm^3$, PC can be used as a repairing material.

The above results confirm that using waste materials like WFS and RHA in PC mixtures was a good idea to enhance the mechanical properties of conventional PCs. Also, such approach reduces the cost of PC, opening a door for more practical and representative applications including use of commercial concrete waste as well.

5.2 Future work

This study has been focused on the main mechanical tests to determine compressive, splitting tensile, and flexural strength. Moreover, several experiments can be done to investigate other mechanical properties.

Hence, the following ideas are suggested:

- Future work must be concerned on tests such as the effect of WFS on impact strength and resistance, chemical resistance under corrosive environments, bond strength determination as a function of curing time, the effect of different temperatures on the curing time as well as freeze–thaw durability of PC which contains WFS as an aggregate.
- Bonding behavior of PC with ordinary concrete could be investigated as well.

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