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https://doi.org/10.1007/s12221-017-6442-2

The Evaluation of the Thermal and Mechanical Properties of Aramid/Semi-Carbon Fibers Hybrid Composites

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Abstract

In this research work, aramid and semi-carbon fibers (SCFs) were hybridized in the form of interlayer or layer by layer into epoxy matrix by hand lay-up method. Afterward, the effect of hybridization on the thermal and mechanical properties of epoxy composites was characterized by thermal analysis; horizontal burning; tensile and bending tests. Based on the results of the mechanical tests, increasing SCFs to aramid fibers ratio decreased tensile strength, elastic and flexural modulus. But with increasing this ratio to 53 % failure strain reduced, whereas in the ratios of more than 53 %, the failure strain enhanced. The results of thermal analysis curves indicated that there are three stage mass loss at the temperature ranges of 100-220, 270-470 and 500-620 ˚C. It was also found that with increasing the SCFs to aramid fibers ratio decreased the third-stage of the mass loss. The results of horizontal burning showed that increasing the SCFs to aramid fibers ratio decreased the rate of burning.

Keywords: Hybrid composite, Aramid fibers, Semi-carbon fibers, Thermal analysis, Mechanical testing

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1- Introduction

It is no secret that aramid fibers are very important reinforcement for advanced composites that the first time were commercially introduced by DuPont in the 1970s under trade name Kevlar [1]. Hence, the Kevlar (polyparaphenylene terephthalamide) is a para-aramid synthetic fiber which is extensively used in civil engineering, automotive and aeronautical industries, owing to the advantage of light weight combined with mechanical and thermal properties [2, 3]. The Kevlar fibers are produced in different types, which are optimized by hot-drawing to develop specific properties. For instance, Kevlar 119 is more ductile than the other types, whereas Kevlar 129 and 149 have the highest strength and elastic modulus, respectively [4]. In this research work, Kevlar 49 was used, which has optimum strength and modulus, as compared with other kinds.

Semi-carbon fibers (SCFs) obtain from the thermal stabilization of polyacrylonitrile (PAN) fibers, which were commercially introduced by R. K. carbon fiber Company under the trade name PANOX. The other name of these fibers is oxidized polyacrylonitrile fibers (OPFs) [5-7]. The most important properties of the SCFs are high thermal resistance, insolubility, high melting point, high electrical resistance, safe biological and ability to absorb moisture [8, 9]. These properties have been led to use the SCFs as fire resistance layer for battery, flame resistance fabrics, surface coating, and feedstock for the production of carbon fibers [10-12]. Based on the reported researches, SCFs with tenacity of 0.2-0.3 GPa, breaking strain of 15-21%, modulus of 7-11 GPa, limited oxygen index of 55% and density of 1.35-1.40 $g.cm^{-3}$ are fabricated by different companies [7, 8].

It is worth noting that influence factors in the properties of the SCFs are chemical composition and the condition of the thermal stabilization of the PAN fibers. In the thermal stabilization, the PAN fibers are heated to be approximately 180-300 ˚C in air atmosphere. During the thermal stabilization, cyclization, dehydrogenization and oxidation reactions occur [13]. Adding comonomers, such as methyl acrylate (MA), sodium methallyl sulfonate (SMS), 2-methyl-2-acrylamidopropane sulfonate (SAMPS) and itaconic acid (IT) are led to increase the rate of the thermal stabilization and the tensile strength of the SCFs. Also, these comonomers reduce the temperature range of the thermal stabilization [14-16]. For these reasons, the chemical composition of the PAN fibers, as feedstocks for the production of the SCFs, consist of 85 wt. % acrylonitrile and 15 wt. % comonomers [17].

In a hybrid composite, two or more types of the fibers are used to reinforce a common matrix such as epoxy, polyester [18]. These fibers can be combined in many different configurations that it can be seen in Fig. 1. In interlayer configuration, see Fig. 1a, the layers of two fibers types are stacked onto each other. The other name of this configuration is layer by layer or laminated. The interlayer configuration is the most common configuration, due to easy to produce. In intralayer configuration, the layers include two or more constituent type of the fibers, which are alternatively arranged within the layers. This is illustrated in Fig. 1b, where different yarns are co-woven into a fabric. Other intralayer configurations such as parallel bundles are also possible [19, 20]. The intralayer configuration are more difficult to produce but has a higher degree of dispersion, as compared with the interlayer configuration. It has been proven both experimentally and theoretically that the higher dispersion of fibers is led to improve the mechanical properties [20-23]. Two fibers types can also be mixed or comingled on fiber level, resulting in an intrayarn configuration that it can be observed Fig. 1c. [19].

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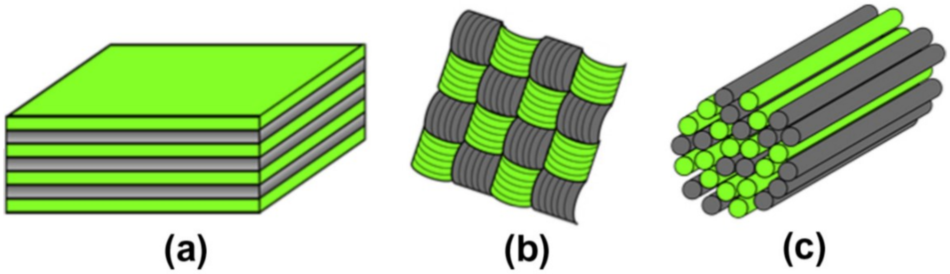

Fig. 1- Hybrid configurations: (a) interlayer, (b) intralayer, (c) intrayarn [19, 20].

It would be worth mention that the SCFs could be hybridized with carbon and glass fibers. Ebrahimnezhad-Khaljiri and Eslami-Farsani studied the effect of hybridization on the thermal and mechanical properties of the SCFs and glass fibers composites. They reported that adding the SCFs into the glass fibers composites caused derivative thermogravimetric (DTG) curves to be shifted to higher temperatures. Also, they found that by increasing SCFs to glass fibers ratio, tensile strength, flexural stress, elastic and flexural modulus were decreased, whereas failure strain of hybrid composites was improved [24].

Based on the best of our knowledge, the aramid fibers can be hybridized with the glass, carbon and the other types of fibers [25-27]. The hybridization of the aramid fibers and SCFs as reinforced into the composites has not been conducted yet. In this regard, the aim of this research work is to study of the thermal and mechanical properties of the aramid fibers-SCFs reinforced hybrid composite, which have been fabricated through hand lay-up method by placing the reinforcements in different stacking sequences.

2- Experimental

2-1- Materials

The epoxy resin used for this study that was the blending of epoxy Bisphenol F resin (ML-506) and polyamine hardener (HA-11) (Mokarrar engineering materials, Iran). The

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resin to hardener ratio was kept to be 100:15 part by weight. The aramid fibers was used for this study is Kevlar 49 (Colan, Australia) in the form of twill 2×2 (175 g/m^2). SCFs with 1.16 dtex linear density (Courtaulds, UK) were used in this study.

2-2- Composite Preparation

The interlayer hybrid composites were fabricated in the form of 4 layer composites through hand lay-up method with the different arranging of the aramid fibers and SCFs reinforcements in the epoxy matrix that it can be seen in Fig. 2. The coding system of these composites listed in Table 1. The resin to reinforcement ratio owing to high absorption resin by SCFs, was selected to be 60:40 wt. %. Curing time for these composites was 24 h at the ambient temperature.

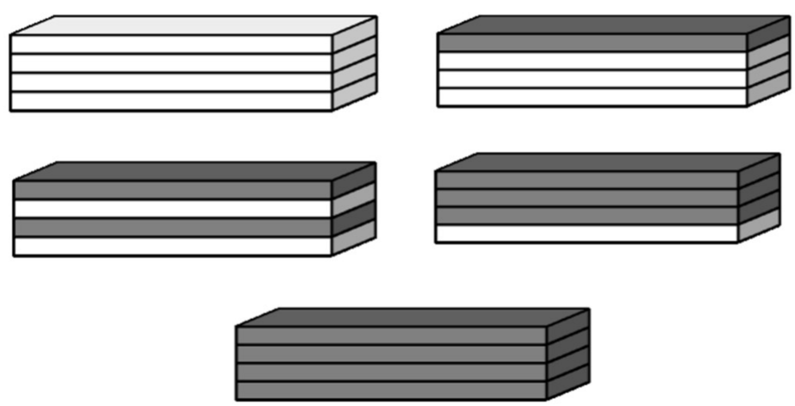

Fig. 2- Different plies of composites, Black layer: SCFs, White layer: aramid fibers.

Table 1- Composites code

Code of composite*	Number of aramid layer	SCFs/total reinforcement ratio (%)
0P-4K	4	0
1P-3K	3	53
2P-2K	2	75

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3P-1K	1	86
4P-0K	0	100

* P: PANOX fibers (SCFs) and K: Kevlar 49 (aramid) fibers.

2-3- Instruments and Characterization

Thermal Analysis

The thermal decomposition of the hybrid aramid fiber-SCFs reinforced epoxy composites was conducted using thermogravimetric (TG) analyzer (TG, NETZSCH TG 209 F1 Iris, Germany). TG tests were performed at the heating rate of 20℃/min and in the temperature range of 50-650 ˚C under nitrogen gas. Considering this point that if the size of samples is too small, the layers of hybrid composites may be delaminated. Therefore the minimum weight of composite samples for TG test was considered approximately10 mg.

Flammability Testing

Horizontal burning test was conducted according to ASTM D 635 with these dimensions: 125 ×12.5 ×2.8 mm^3 (length × width × thickness) [28]. For more accuracy, at least three samples were examined and average value was reported.

Tensile Test

The tensile test of the composites was performed by Hounsfield H25KS tester to load up 25 kN and cross head 2 mm/min. The samples were cut and polished with the dimension of 250×20 mm^2 according to ASTM D3039 [29]. The gauge length was set 100 mm and abrasive papers P80 were used to its ends for gripping and ensuring failure within the gauge region. For more accuracy, at least five samples were examined and the average value was reported.

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Flexural Test

The flexural test of the composites was performed by Hounsfield H25KS tester with 500 N load cell. The flexural performance was measured in three point bending mode, according to ASTM D790 method. The samples with the dimension of 100×20 mm^2 were cut and polished. Support span length and cross head speed were set to be 80 mm and 4.3 mm/min, respectively, according to ASTM D790 [30]. For more accuracy, at least five samples for each composite were tested and the average value was reported.

3- Results and Discussion

3-1- Thermal Analysis of Composites

Figs. 3 and 4, present the TG and derivative thermogravimetric (DTG) curves for the hybrid aramid fibers-SCFs reinforced epoxy composites with the different layering sequence, respectively. Table 2, summarizes some data extracted from TG and DTG curves which included decomposition temperature recorded at 5% mass loss ($T_{5\%}$), the first onset decomposition temperature (T_{o1}), the second onset decomposition temperature (T_{o2}), the temperature of the maximum rate of the mass loss (T_{max}) and char residue at 650 ˚C.

Considering Fig. 3 and Table 2, 2P-2K composite with 292.5 ˚C had the maximum decomposition temperature at 5 % mass loss. Also, from Fig. 3, it could explicitly be observed that 0P-4K composite with 20.2 % char residue had the minimum char residue in nitrogen atmosphere at 650 ˚C. It was also realized that 2P-2K composite with 43.31 % char residue had the maximum char residue. The reason of this behavior was to difference in the rate of the mass loss and heat transfer between the layers of the reinforcement, which have been led to 2P-2K composite had the maximum char residue.

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Based on Fig. 4, it could be seen that there were three stage of the mass loss in the DTG curves of hybrid composites except 4P-0K composite which had the 2 stage of the mass loss. The first mass was loss at the temperature range of 100-220 ˚C that mainly caused by the pendant chain of epoxy, whereas second mass loss resulted at the temperature range of 270-470 ˚C that owing to the decomposition of the main chain in epoxy resin [31]. From Fig. 4, it could be understood that 0P-4K and 3P-1K composites with 320 ˚C and 280 ˚C T_{o1} had the maximum and minimum T_{o1}, respectively. Considering to these DTG curves, it could be understood that increasing the SCFs to aramid fibers ratio, the first onset decomposition shifted to the lower temperatures. This is shows that additionally, the decomposition of the main chain in epoxy resin, there is another factor that has important role in the mass loss. When SCFs are placed under nitrogen atmosphere at high temperatures, the necessary conditions are created for converting the SCFs to carbon fibers. During this converting is performed carbonization reactions and some of the materials are signed out in the form of NH_3 and HCN gases [32]. Consequently, the exhaust of these gases are caused that with increasing the SCFs to aramid ratio, the DTG curves shifted to lower temperatures.

Based on Fig. 4, it could be clearly observed that there is another mass loss at the temperature range of 500-620 ˚C due to the decomposition of the aramid fibers [33]. By increasing the SCFs to aramid fibers ratio, this mass loss shifted to the higher temperatures. As already mentioned, during TG test in the nitrogen atmosphere, carbonization reactions and forming carbon fibers occur. Forasmuch as, the carbon fibers have high thermal resistant, it can assume that the formed carbon fibers have reduced the rate of heat transfer.

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Consequently, with increasing the SCFs to aramid fibers ratio, the decomposition and mass loss temperature of aramid fibers were shifted to the higher temperatures.

In light of the above, it could be mentioned that SCFs have a negative effect in hybrid composites in nitrogen atmosphere at low temperature, owing to carbonization reactions, whereas these fibers have a positive effect at high temperatures in fabricated hybrid composites, due to formed carbon fibers in nitrogen atmosphere.

Table 2- Thermal properties of hybrid composites recorded from TG and DTG

Code of composite	T_{max} (℃)	$T_{5\%}$ (℃)	T_{o1} (℃)	T_{o2} (℃)	Char residue at 650℃ (%)
0P-4K	400.7	286.0	320.0	510.0	20.20
1P-3K	383.0	266.0	314.0	552.0	30.72
2P-2K	364.5	292.5	286.5	544.5	43.31
3P-1K	366.0	240.0	280.0	563.0	41.30
4P-0K	370.7	255.7	290.6	——	29.16

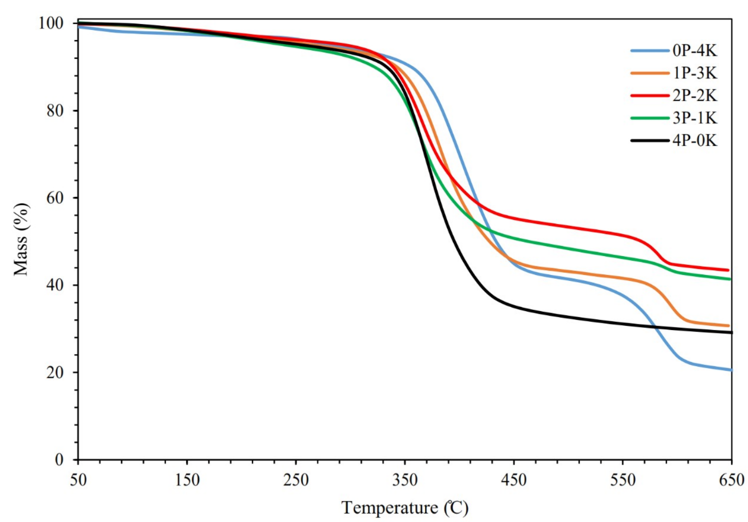

Fig. 3- TG curves of SCFs-aramid fibers hybrid composites.

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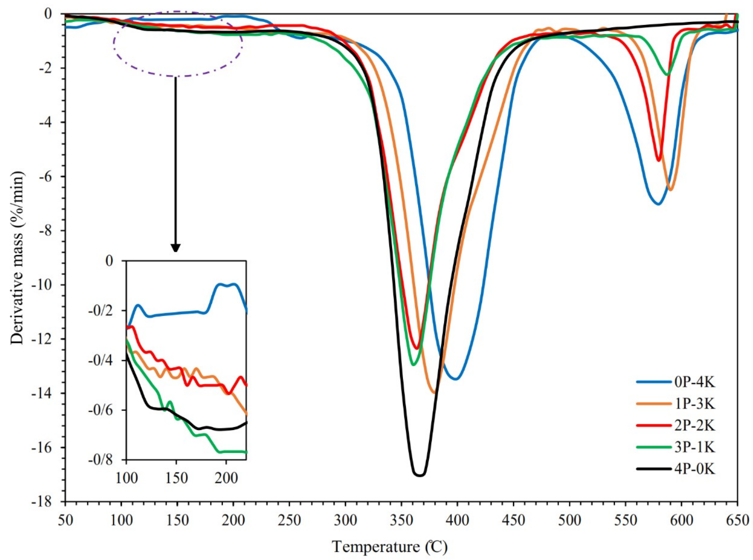

Fig. 4- DTG curves of SCFs-aramid fibers hybrid composites.

3-2- Flammability Testing of Composites

The results of horizontal burning technique have been listed in Table 3. Fig. 5 shows burning rate-SCFs to total reinforcement ratio curve for hybrid aramid fibers-SCFs reinforced composites. As shown in Fig. 5, 0P-4K and 4P-0K composites with 30 and 8.13 mm/min rate of burning had the maximum and minimum rate of burning, respectively. The obtained results showed that increasing the SCFs to aramid fibers ratio decreased the burning rate of hybrid composites consequently increased the flame retardancy of the hybrid composites due to the chemical structure of the SCFs which could be seen Fig. 6 [5]. From Fig. 6, it could be understood that firstly, the SCFs had the 40% oxidized structure and secondly, for accomplishment of the burning reactions must be broken the chains of the SCFs. The chain scission of the SCFs have to need high level of energy. For this reason, SCFs is flame retardant and adding to the epoxy composites are caused flame retardancy.

Table 3- Horizontal burning of composites

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Code of composite	0P-4K	1P-3K	2P-2K	3P-1K	4P-0K
Rate of burning (mm/min)	30.00±0.55	20.31±0.45	14.85±0.35	12.13±0.40	8.13±0.25

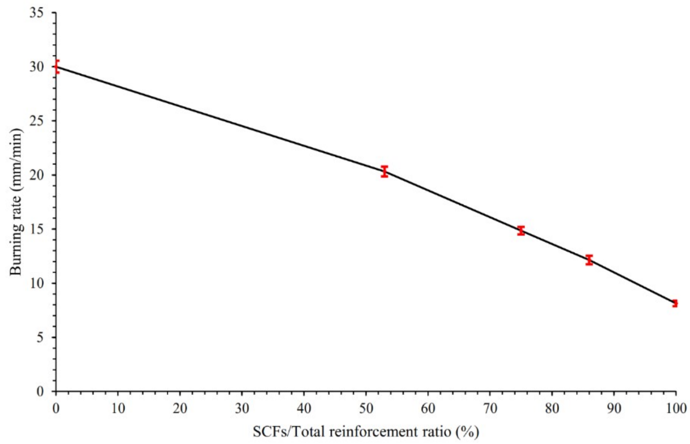

Fig. 5- Burning rate of SCFs-aramid fibers hybrid composites.

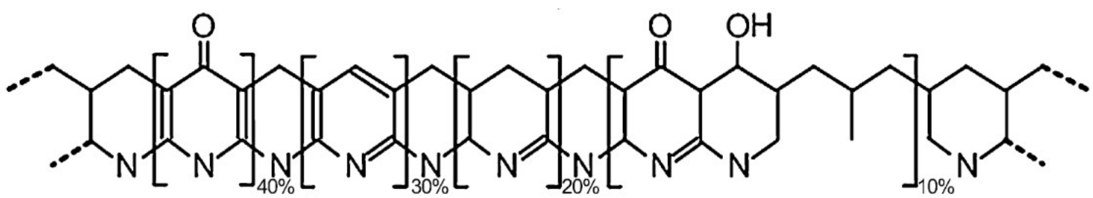

Fig. 6- Chemical structure of stabilized PAN [5].

3-3-Tensile Test of Composites

Fig. 7, depicts the average stress-strain curves of the hybrid aramid-SCFs reinforced composites. Table 4, summarizes some data extracted from these curves that included tensile strength, elastic modulus and failure strain. Fig. 8, presents the tensile strength-SCFs to total reinforcement ratio curve that has been extracted from stress-strain curves. From this curve, it could be seen that 0P-4K and 4P-0K composites had the maximum and minimum tensile strength, respectively. Results showed that increasing the SCFs to aramid ratio, decreased the tensile strength due to the low tensile strength of the SCFs. The reason

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of the low tensile strength of the SCFs is to the formation of weak crosslinks between the ladder structures of the SCFs molecules, during thermal stabilization process. When SCFs were set under tension stresses, these crosslinks would be broken consequently the SCFs would be failed [34].

Table 4- Results of tensile test

Code of composite	Tensile strength (MPa)	Elastic modulus (GPa)	Failure strain (%)
0P-4K	322.44 ± 10.01	19.60 ± 0.90	1.75 ± 0.06
1P-3K	152.37 ±8.51	11.86 ± 0.56	1.22 ± 0.03
2P-2K	108.03 ± 8.32	8.74 ± 0.36	1.23 ± 0.06
3P-1K	73.69 ± 3.63	6.41 ± 0.27	1.28 ±0.03
4P-0K	61.07 ± 6.73	4.87 ± 0.06	1.68 ± 0.04

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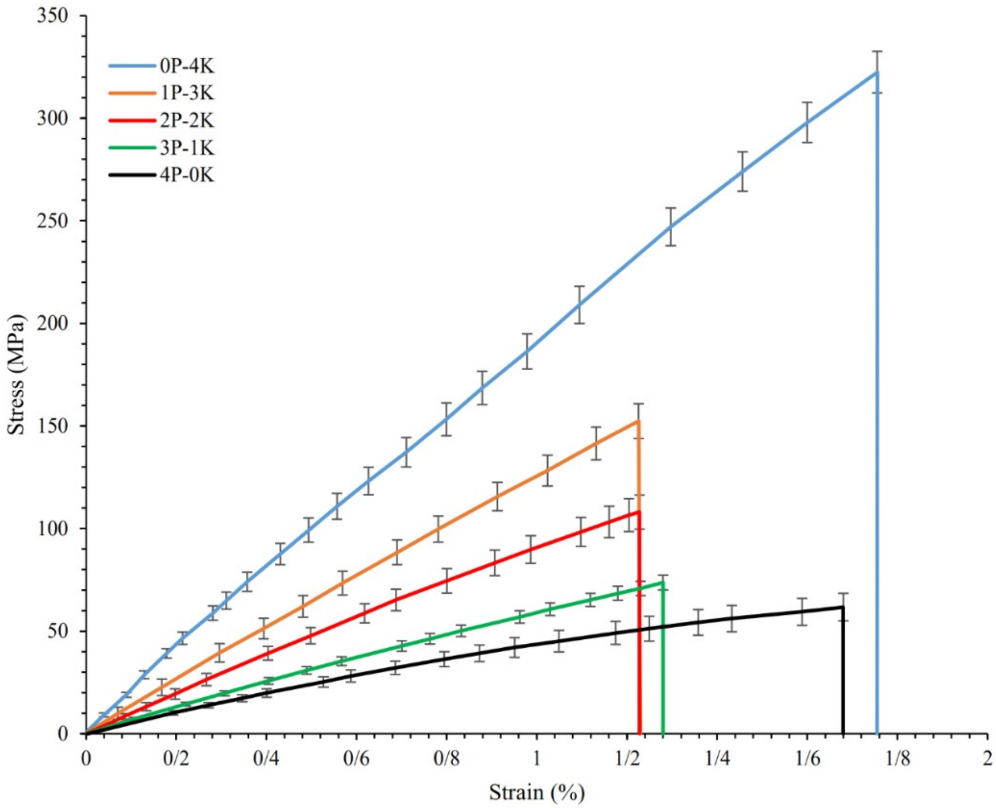

Fig. 7- Stress-Strain curves of SCFs-aramid fibers hybrid composites.

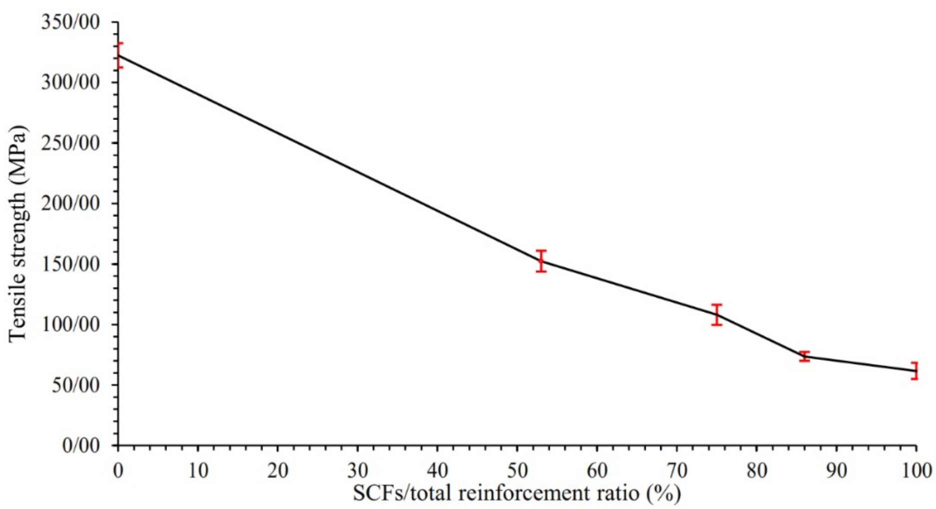

Fig. 8- Tensile strength of SCFs-aramid fibers hybrid composites.

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Fig. 9, demonstrates the elastic modulus-SCFs to total reinforcement ratio curve that has been extracted from stress-strain curves. This curve had similar behavior like the tensile strength-SCFs to total reinforcement ratio curve. From this curve, it could be shown that 0P-4K composite with 19.60 GPa and 4P-0K composite with 4.87 GPa elastic modulus, had the maximum and minimum elastic modulus, respectively. Results showed that increasing the SCFs to aramid ratio decreased the elastic modulus owing to the low elastic modulus of the SCFs.

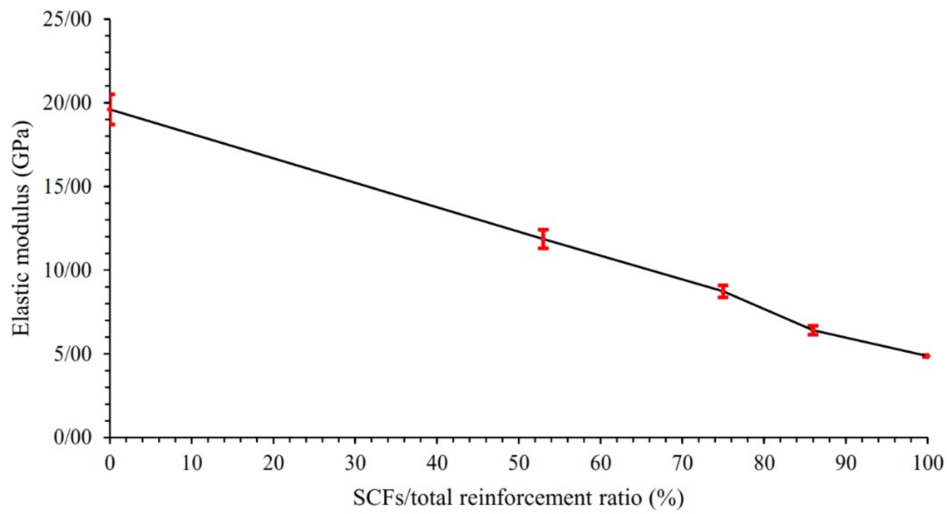

Fig. 9- Elastic modulus of SCFs-aramid fibers hybrid composites.

Fig. 10, shows the failure strain-SCFs to total reinforcement ratio curve that has been extracted from stress-strain curves. Results of data indicated that 0P-4K and 1P-3K composites had the maximum and minimum failure strain, respectively. Based on this curve, it could be seen that increasing the SCFs to aramid fibers ratio, the failure strain initially decreased and then increased. The factors of uniformity between two fibers reinforcement and residual stresses have important role to the creation of this behavior [19].

If the sequence of the fibers (aramid-SCFs) in composite, in such a way that created the higher level of mismatch, is caused delamination and debond in the interfaces of the fibers during the tensile test. For this reason, 1P-3K composite had the minimum failure strain. In other sequence with reducing the mismatch reduced the effect of the uniformity factor and the residual stress is only effective factor. Based on researches, the fibers have different thermal expansion coefficient. The residual stress is created after curing and during cooling processes. In practice, resin with suitable bonding between the aramid fibers and SCFs is caused that changing the length of the fibers didn't happen. This is caused that the compressive stresses is created on the fibers with lower elongation. These compressive stresses counteract the effect of the tensile stresses from tensile test, consequently the failure strain (maximum elongation per primary length) of the fibers with lower elongation increases. In the same way, the creation of the tensile stresses on the fibers with higher elongation are caused reducing the failure strain [19]. Interaction between the apparent failure strains depends on the fibers ratio in the hybrid composites that based on Fig. 10, with increasing the SCFs to aramid fibers ratio in higher percentage than 53% increases the failure strain.

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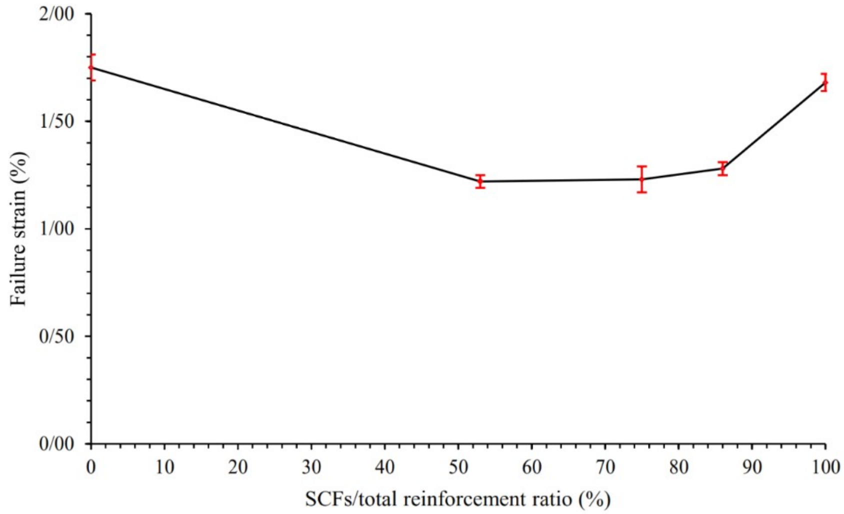

Fig. 10- Failure strain of SCFs-aramid fibers hybrid composites.

3-4- Bending Test of Composites

The data obtained from bending test for hybrid aramid fibers-SCFs reinforced epoxy composites containing flexural modulus and flexural stress have been listed in the Table 5, and have been visualized in Figs. 11 and 12, respectively. This data has been calculated according to standard test method ASTM D790 [30]. It should be mentioned that the crosshead of machine was placed on the SCFs layers in all hybrid composites, owing to the creation of pressure stresses on SCFs layers.

The flexural modulus had the similar behavior like the elastic modulus, see Figs. 9 and 11. Therefore, 0P-4K composite with 16.22 GPa and 4P-0K composite with 4.78 GPa flexural modulus, had the maximum and minimum flexural modulus, respectively. Increasing the SCFs to aramid fibers ratio decreased the flexural modulus due to low flexural modulus of SCFs. From Fig. 12, it could be observed that increasing the SCFs to aramid fibers ratio up to 75 wt. % increased the flexural stress. But more than this percent decreased the flexural stress. This is because of the ladder polymer structure of the SCFs

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that under the tensile stresses are weak, whereas under the compressive stresses have good resistance [5, 6]. Based on, the different sequence of the SCFs and aramid fibers in Fig.2, it could be assumed that during the bending test, the compressive and tensile stress are applied on the SCFs and aramid fibers, respectively, consequently, the failure strain up to 75 wt. % (2P-2K) increased. But with increasing the layering of the SCFs, the lower layers are to under the tensile stresses, consequently the failure strain reduced.

Table 5- Results of flexural test

Code of composite	Flexural stress (MPa)	Flexural modulus (GPa)
0P-4K	105.93 ± 7.62	16.22 ± 0.42
1P-3K	187.37 ± 8.00	10.76 ± 0.51
2P-2K	198.52 ± 5.75	8.10 ± 0.53
3P-1K	178.79 ± 6.85	6.24 ± 0.49
4P-0K	107.97 ± 5.59	4.78 ± 0.33

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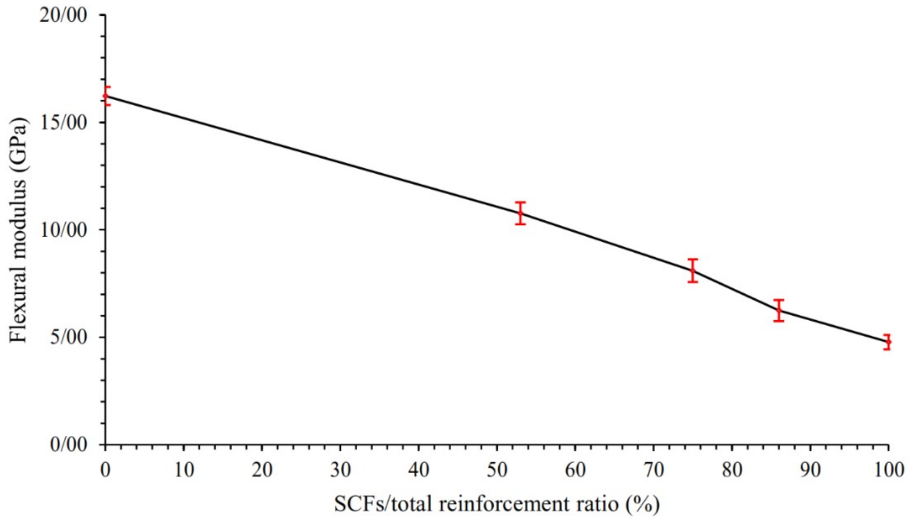

Fig. 11- Flexural modulus of SCFs-aramid fibers hybrid composites.

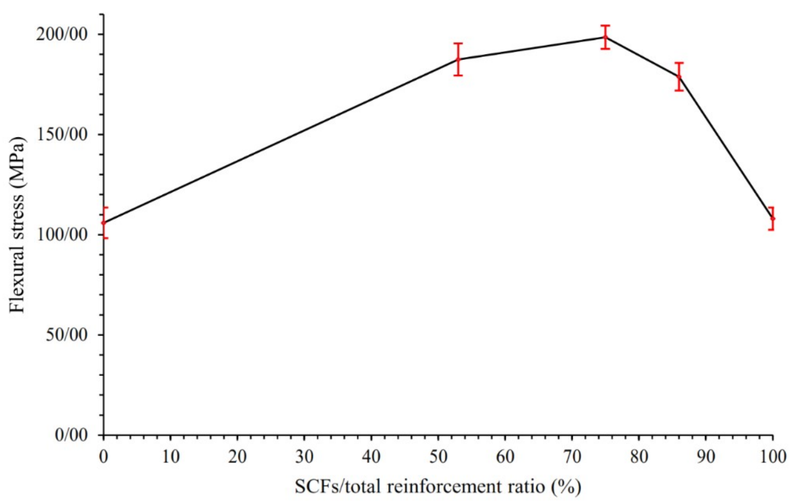

Fig. 12- Flexural stress of SCFs-aramid fibers hybrid composites.

4- Conclusions

In this research work, semi-carbon and aramid fibers were combined in interlayer configuration with different stacking sequence into epoxy matrix. Afterward, thermal and mechanical properties of these composites were studied. The following conclusion could be drawn based on the results above:

1. The results of thermal analysis indicated that there are three stage of mass loss. In the second stage, increasing semi-carbon fibers to aramid fibers ratio, the temperatures of the maximum rate of the mass loss shifted to lower temperatures but in the third stage with increasing the semi-carbon fibers to aramid fibers ratio reduced the decomposition of the aramid fibers.
2. The result of horizontal burning showed that increasing the semi-carbon fibers to aramid fibers ratio decreased the rate of burning thereby increased flame retardancy.
3. The results of tensile test depicted that increasing the semi-carbon fibers to aramid fibers ratio decreased tensile strength and elastic modulus. But failure strain at beginning decreased and then increased.
4. The results of bending test demonstrated that increasing the semi-carbon fibers to aramid fibers ratio decreased flexural modulus, whereas flexural stress at first increased and then decreased.

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This is the accepted manuscript (postprint) of the following article:
Ebrahimnezhad-Khaljiri, H., Eslami-Farsani, R., & Banaie, K. A. (2017). The evaluation of the thermal and mechanical properties of aramid/semi-carbon fibers hybrid composites. *Fibers and Polymers*, *18*(2), 296-302.
https://doi.org/10.1007/s12221-017-6442-2

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https://doi.org/10.1007/s12221-017-6442-2

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