FACULTY OF TEXTILE ENGINEERING TUL

Carbon-based functional structures from pyrolysis of Kevlar Fabric Wastes

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SUMMARY OF THE THESIS

Title of the thesis:	Carbon-based functional structures from pyrolysis of Kevlar Fabric Wastes
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Mode of study:	Full time
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Abstract

This dissertation is dealing with the use of fibrous aromatic polyamide (Kevlar) waste for the development of microporous and electrically conductive materials based on activated carbon. A method of controlled, one-stage carbonation is used.

The waste Kevlar fabric was obtained from a regional manufacturing plant in the Czech Republic. Activated carbon structures were prepared by pyrolysis under different conditions, i.e. several types of inert atmosphere, optimized time-temperature mode of heat stress, and final carbonization temperature (in the range of 500 °C to 1200 °C) so as to create porous and electrically conductive structures.

The thermal degradation of Kevlar and the composition of volatile products of its pyrolysis were investigated.

The geometric, physical, morphological, electrical, and thermoelectric properties of the prepared activated carbon structures were studied with respect to different thermal modes and different types of inert atmospheres. The electromagnetic interference (EMI) shielding capability in the high frequency (i.e. at 2.45 GHz) and low frequency (i.e. below 1.5 GHz) regions was investigated using the waveguide method and the coaxial line transmission method. Furthermore, the ohmic heating behavior of activated carbon structures was studied as a function of the applied electric power and time.

Progressive changes in concentrations of gaseous products of thermal decomposition of Kevlar depending on the pyrolysis temperature and their differences in volatility were evaluated using an algorithm for the separation of mixed spectra obtained by UV spectroscopy.

Keywords: Kevlar, pyrolysis, activated carbon, carbonization, graphitization, organic volatile compounds, spectral matrix decomposition, joule heating, electromagnetic shielding.

Abstrakt

Tato disertační práce se zabývá využitím vlákenných aromatických polyamidových (Kevlarových) odpadů pro vývoj mikroporézních a elektricky vodivých materiálů na bázi aktivovaného uhlíku. Je použita metoda řízené, jednostupňové karbonizace.

Odpadní Kevlarové textilie byl získány z regionálního výrobního závodu v České republice. Aktivované uhlíkové struktury byly připraveny pyrolýzou za různých podmínek, tj. několika typů inertní atmosféry, optimalizovaného časově teplotního režimu tepelného namáhání a finální teploty karbonizace (v rozmezí 500 °C až 1200 °C) tak, aby vznikly porézní a elektricky vodivé struktury. Byla zkoumána tepelná degradace Kevlaru, a složení těkavých produktů jeho pyrolýzy.

Geometrické, fyzikální, morfologické, elektrické a termoelektrické vlastnosti připravených aktivovaných uhlíkových struktur byly studovány s ohledem na různé tepelné režimy a různé typy inertní atmosféry. Schopnost stínění proti elektromagnetickému rušení (EMI) ve vysokofrekvenčních oblastech (tj. na 2,45 GHz) a nízkofrekvenčních oblastech (tj. pod 1,5 GHz) byla zkoumána pomocí metody vlnovodu a metody koaxiálního přenosu. Dále bylo studováno chování ohmického ohřevu aktivovaných uhlíkových struktur jako funkce použitého elektrického výkonu a času.

Progresivní změny koncentrací plynných produktů tepelného rozkladu Kevlaru v závislosti na teplotě pyrolýzy a jejich rozdíly v těkavosti jsou vyhodnoceny pomocí algoritmu pro separaci směsných spekter získaných UV spektroskopií.

Klíčová slova: Kevlar, pyrolýza, aktivovaný uhlík, karbonizace, grafitizace, těkavé produkty pyrolýzy, rozklad matice UV spekter, ohmický ohřev, elektromagnetické stínění.

Summary

1. Introduction

Climate change and environmental issues continue to shape society's view of sustainability, circular economy, and recyclable/reusable items [1]–[3]. The growing demand for textiles has increased waste production during manufacture and end-of-life, compounding disposal concerns. The world generates 92 million tons of textile waste each year, and only 15% of it gets recycled [4], [5]. 25% of the 5.8 million tonnes of textiles discarded in the EU are recycled [6]. Europe generates 15 kg of textile waste per person, and apparel and footwear consumption is expected to rise 63% by 2030 [7]. Existing approaches to lowering discarded polymers are not enough to address the build-up of polymeric wastes [8]. Regardless of how polymeric fibrous materials have improved the standard of our living, it is tough to manage fibrous wastes. Existing approaches to lowering discarded polymers are not enough to address the build-up of polymeric wastes [8]. Recycling, landfills (including discarded ones), and incineration are all options for managing polymeric fibrous wastes. However, toxic gases are produced when polymers are burned. Additionally, conventional recycling has drawbacks because the recycled polymer has diminished durability and flexibilityThe most ingenious way to manage fiber wastes is "upcycling" into solid carbonaceous materials. Upcycling involves pyrolysis (>250 °C) and carbonization (>800 °C) in an inert environment at a variable heating rate to produce carbon-rich materials [9].

1.1.Upcycling of Polymeric wastes to Carbon-based functional materials

Carbonized materials derived from upcycling of polymeric fiber wastes are consumed for versatile applications such as adsorbents in waste purification, energy and the environment, and electrical energy storage (EES) systems. The purpose of the upcycling process, as indicated in Fig. 2, is to produce carbon-rich solid wastes rather than oils or volatile chemicals.

Fig. 1. Upcycling of Fibrous wastes to Carbon-based functional materials. Recreated from [9].

The development of carbonaceous materials is influenced by factors such as the nature of the precursor used, the application of activating agent, and emphatically the process conditions of pyrolysis. The operational parameters in the pyrolysis process have a discernable effect on the resulting manner of decomposition and on the final characteristics of the product [10]–[12].

1.2.Carbon-based functional materials

Bonding (hybridization of carbon atom orbitals) and dimensionality (non-nanoscale dimensions) classify the most frequent carbon materials. Fig. 3 shows carbonaceous minerals in all dimensions [13]. Activated carbon fibers, mesoporous carbon, graphite, graphene, CNTs, fullerenes, and carbon quantum dots are a selection of carbon-based materials derived from fibrous sources [14]–[16]. Activated carbon fiber (ACF) has a fiber shape and a clearly defined porosity structure, making it a viable microporous material. High packing density, outstanding volumetric capacity, rapid adsorption/desorption, and low handling effort are just a few of ACF's unique properties. Because of their exceptional structural (physical and chemical), thermal (heat transfer), and electric qualities, carbon-based materials may be useful in a broad spectrum of applications [17].

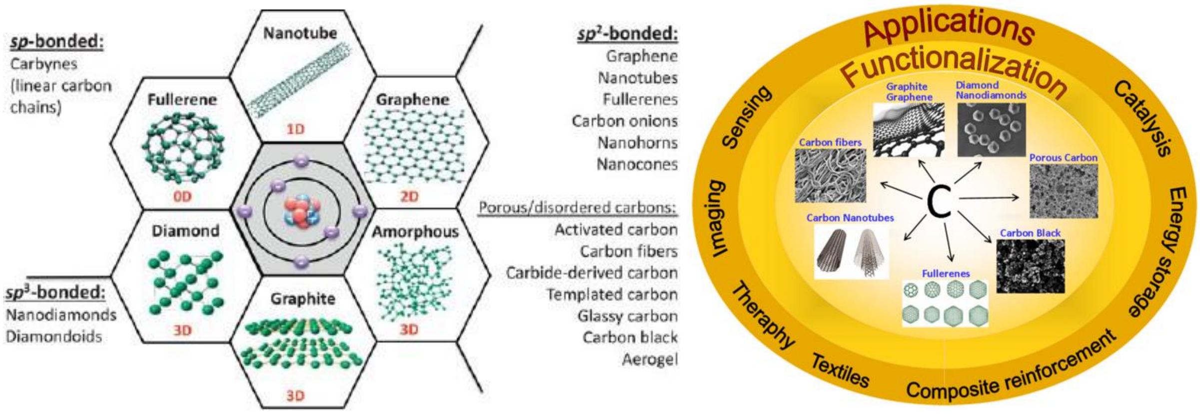


Fig. 2. Classification of carbon-based materials and potential applications [18], [19].

1.3.Manufacturing process of ACF’s

The prevailing steps involved in the production of ACF’s mainly include the following stages (as shown in Fig .5): stabilization, pre-treatment for-instance, acid impregnation (prior to carbonization), carbonization/pyrolysis, and in some cases activation (physical or chemical activation). However, it must be mentioned that pre-treatment and stabilization steps depend on the type of precursor and are not necessarily included in the preparation of ACF.

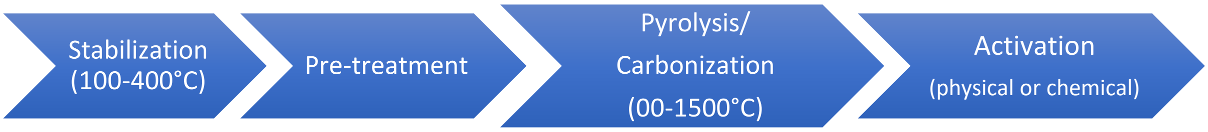


Fig. 3. Main stages involved in the production of ACFs.

In the stabilization phase, controlled low-temperature heating in air at a temperature between 200 and 300 °C stabilizes the precursor. This procedure, which is essential for producing high-quality carbon fibers, may take several hours, depending on the temperature, precursor diameter, and precursor fiber properties. By thermal decomposition at high temperatures and under the direct flow of inert gas, carbonization converts the initial component into a carbonized substance. Fibers that have been thermally stabilized are carbonized in an inert environment that contains gases like nitrogen (N2) or argon (Ar). In most cases, the type of application for the produced carbon fibers determines the carbonization temperature [20], [21].

1.4.Types of precursors

The ideal features of precursors required to manufacture carbon fibers are easy conversion to carbon fiber, high carbon yield, and cost-effective processing [22]. The usual preference as precursors for activated carbon materials is low-crystallinity feed-stocks like polyacrylonitrile, phenolic resins, coal tar pitch, etc. The precursor materials of the carbon fibers are important because the combination of various properties and behaviors (mechanical, physical, and chemical) potentially required [23], [24]

Following are some of the prevalent and widely used choices of precursors for carbon production:

i. Polyacrylonitrile (PAN) precursors: More than 80% of the worldwide carbon fiber market is accounted for by goods based on polyacrylonitrile (PAN). Following PAN-based carbon fiber are pitch-based carbon fiber, rayon-based carbon fiber, and polyolefin-based carbon fiber. Most industrial firms have been successfully preparing carbon fiber with PAN precursors for many years now [25], [26].

ii. Cellulosic precursors: The utilization of cellulose precursors has been discovered to be advantageous in the production of carbon fibers due to the thermal decomposition of cellulose without undergoing a melting process and the existence of a highly organized crystalline configuration. They contain 44.4% carbon, although in reality, the reaction is more involved than simple dehydration, thus the yield of carbon is only about 25–30% [15].

iii. Pitch-based precursors: Pitch-based precursors are complex mixtures of polyaromatic molecules and heterocyclic compounds that have a high yield (85%), and the resulting carbon fibers have a high modulus due to their graphitic nature. Pitch-based carbon fibers, on the other hand, exhibit inferior compression and transverse properties than PAN-based carbon fibers [27].

iv. High-performance fibrous wastes precursors: These precursors are beneficial mainly due to well ordered oriented structure with a higher portion of carbon. One of the most useful representants is aromatic polyamides (aramids).

Aramids are full aromatic polyamides, which are long-chain aromatic polyamides where the amide connections are connected to two aromatic rings to the extent of at least 85% [28]. As a polyamide synthesized by condensation, poly(p-phenylene terephthalamide) (PPTA), prevalently known as Kevlar® (hereafter referred to as Kevlar), are fully crystalline with a small fraction of randomly oriented material and Fig. 1 depicts the chemical structure.

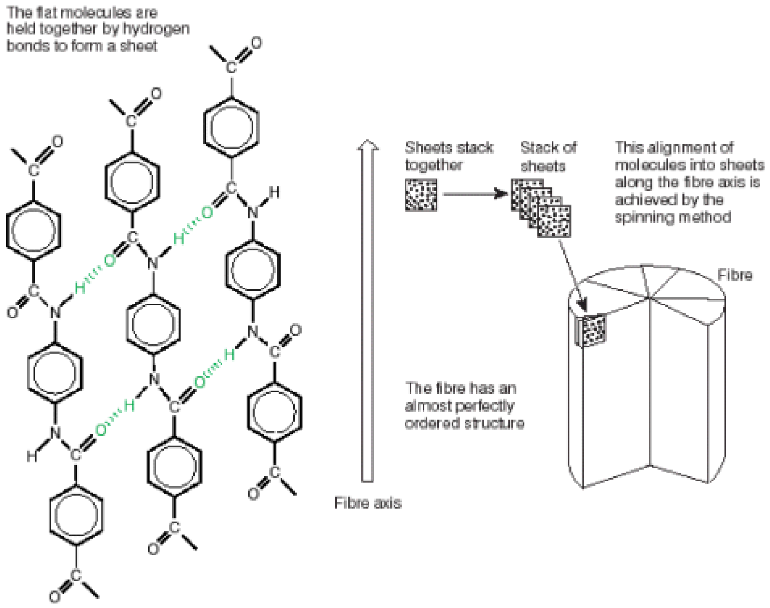


Fig. 4. Structure of Kevlar [29].

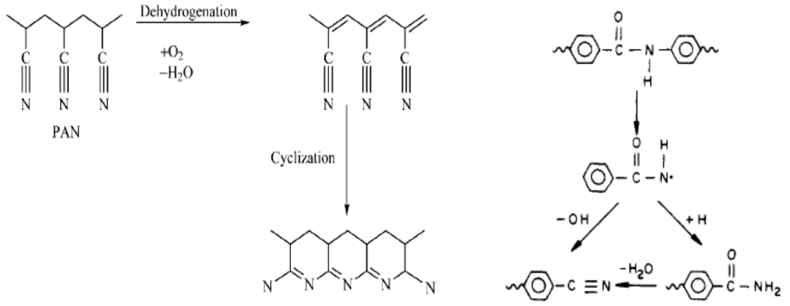
Freeman et al. [30] first used Kevlar fibers as precursor materials to obtain carbons with high surface areas, narrow pore size distributions, and acceptable yields and mentioned homogeneous pore size distributions of obtained activated carbon fibers [31], [32]. Later, a number of studies focused on the preparation of activated carbon fibers from Kevlar and Nomex fibers were proposed [33]–[37]. More substantially, the oxidative stabilization phase that is frequently necessary for the carbonization of low melting precursors can be avoided by taking advantage of Kevlar fibers' excellent thermal stability. Additionally, as Kevlar flocks are leftovers from the manufacture of the material, their lower price may be a benefit if they are used as the precursor [38].

Alternatively, over the years, a trend has emerged for the production of low-cost, useful carbonaceous materials from various types of precursors such as regenerated cellulose [15], [24], [39], Polyethylene terephthalate (PET) [40], [41], Polyethylene (PE) [42]–[44], Polypropylene (PP) [45], polyamides [36], polyaramids [46], polybenzobisoxazole (PBO) [47], polyvinylidene fluoride (PVDF) [48], poly(vinylacetylene) [49], polyvinyl alcohol [50], and phenolic resins [51] have been investigated for manufacturing carbon fibers.

2. Purpose and the aim of the thesis

The augmented demands of textile materials over time have brought challenges in the disposal of substantial volumes of waste generated during the processing and end-of-life of such materials. Taking into consideration environmental safety due to the discarding of textile waste, it becomes critical to recuperate useful products from such waste for economic reasons. In contrast to the conventional recycling mechanism, the most innovative and cutting-edge approach to managing fibrous wastes is ‘upcycling’ into solid carbonaceous materials. The primary method of upcycling is carbonization, or pyrolysis, which calls for high heat (>800 °C), a variable heating rate, and an inert environment. Pyrolysis involves the elimination of moisture and volatile components from fibrous materials by cleavage of molecular bonds to finally obtain carbon-rich residues. Over the years, there have been numerous studies on the pyrolysis of poly (1,4 phenylene terephthalamide) (Kevlar) and the characterization of volatile products by using various analytical techniques.

Carbon-based materials are typically produced using a range of complex and multistep procedures from semi-crystalline precursors such as viscose rayon, phenolic resins, polyacrylonitrile (PAN), or isotropic coal tar pitches. This results in a higher production cost due to the requirement for thermal stabilization (in air at temperatures ranging from 200 to 300 ºC). Dehydrogenation, oxidation, and nitrile cyclization reactions occur during this process (as shown in Fig .2a), which is followed by carbonization in an inert environment at temperatures ranging from 500 to 3000 ºC. However, due to the rigid-rod structure and high degree of aromaticity, direct carbonization is applicable in the case of Kevlar in order to obtain carbon. Progressive decomposition of amide groups occurs at temperatures between 550 and 575 ºC, resulting in the formation of intermediate aryl nitrile species. (shown in Fig .2b)



(a) (b)

Fig .2. Chemical process of producing carbon materials from (a) PAN-based precursor by two-stage carbonization and (b) Kevlar-based precursor by direct carbonization.

There is a growing demand for more effective and eco-friendly advancements in Electrical conductivity, electromagnetic interference (EMI) shielding, and Joule heating performance of textiles in particular, which have compelled much attention in various applications. The porous morphology, large specific surface area, and higher electrical conductivity of materials are important factors for effective EMI shielding and joule heating. Because of their inherent thermal and electrical qualities, carbonaceous materials in various forms (fibers, textiles, or particles) have received substantial attention as effective heating elements. This has a wide range of applications, including temperature and current sensors, grounding materials to dissipate static charge, structural health monitoring, deicing in aerospace and wind turbine constructions, electromagnetic interference shielding, and so on.

The principal objectives discussed here are:

- Development of porous and electrically conductive activated carbon by single-stage carbonization and physical activation of Kevlar fabric under varying conditions of temperature, rate of heating, and pyrolysis atmosphere, without any intermediate stabilization step and holding time.
- Investigation of the carbonization of Kevlar fabric by three different methods.
- Characterization of morphology, physical, chemical, electrical, and thermoelectric properties of activated carbon fabrics.
- Description of progressive changes in concentrations of decomposition products as a function of pyrolysis temperature and their difference in volatility, by using an algorithm for separation of mixture spectra obtained by UV spectroscopy.
- Evaluation of surface and volume electrical resistivity, EMI shielding effectiveness of activated carbon fabrics, and the Joule heating behavior of the activated carbon fabrics.

3. Overview of the current state of the problem

3.1.Theory of Pyrolysis and Carbonization

The term "Pyrolysis" is frequently limited to high-temperature reactions that only produce gases and char, but it also refers to the thermolysis of organic macromolecules at temperatures between 250 and 800 °C (in the absence of oxygen) that produces gaseous, liquid, and solid byproducts. The carbonization phase, following pyrolysis, is realized at 800°C and is characterized by fundamental variations in the chemical and physical properties of the organic material and is irreversible [3].

The first stage of the pyrolysis process involves an intramolecular endothermic chemical reaction that either involves the rearrangement of chemical bonds followed by the elimination of small molecules, or the cleavage of chemical bonds followed by the stabilization of the unstable fragments (the former reactions take place at around 300 °C, and the latter above 500 °C [30], [52], [53]. Under the influence of heat, hetero atoms (oxygen and hydrogen) are removed, and the substance becomes richer in carbon. The remaining carbon atoms form aromatic sheets with a certain planar structure. These sheets are then placed in a random manner, creating spaces between them, resulting in the pyrolyzed product's principal porosity [54]. In general, polymers breakdown to a primary char and water below 550°C. Further heating above 550°C gradually converts the primary char to a conglomerate of loosely linked small graphitic regions that is virtually independent of the structure of the original polymers [55].

Numerous parameters affecting the structure and properties of carbonizates influence the mechanism of carbonization of macromolecular compounds and the qualities of the final carbon products. These include the initial polymer preparation approach, heat treatment conditions, the presence of additional chemical substances in the environment of thermochemical reactions, and, in general, specific attributes of the carbon material preparation process [56], [57].

The process of thermal decomposition of materials is of major significance to their structural and thermal properties. Furthermore, the products of the decomposition of a material may play a vital role with regard to great temperatures or fire exposure [52], [53], [58]–[60]. Therefore, it is of prime importance to investigate the thermal decomposition process of a polymeric material. Early investigations on the pyrolysis of Kevlar were based on chemical analysis and the study of volatile degradation products, which revealed that carbon was acquired through non-isotropic and non-graphitizable structures [61]–[64].

3.2. State of the Art in Kevlar Pyrolysis

A pyrogram of evolved gasses during thermal degradation (at 20 °C/min heating rate) of Kevlar shows that most of the degradation products are released from 550 °C – 650 °C [64], [65]. The volatiles evolving from Kevlar pyrolysis is constituted of various aromatic products with one, two, or three rings. These compounds were frequently formed from Ph-CO and HN-Ph (phenyl group: Ph) bonds cleavage and mainly contained amino and nitrile substituents. Both homolytic and heterolytic reactions are involved in the formation of the thermal degradation products of aromatic polyamides. At 400 °C, Kevlar pyrolysis mostly produces H_2O and CO_2, according to Krasnov et al. [15], who similarly came to the conclusion that the thermal degradation mechanism involves hydrolysis of the NH-CO bonds and subsequent decarboxylation of carboxyl end groups. a tentative degradation pathway accounting for the formation of radicals in the polymer is shown in Fig .8 [53].

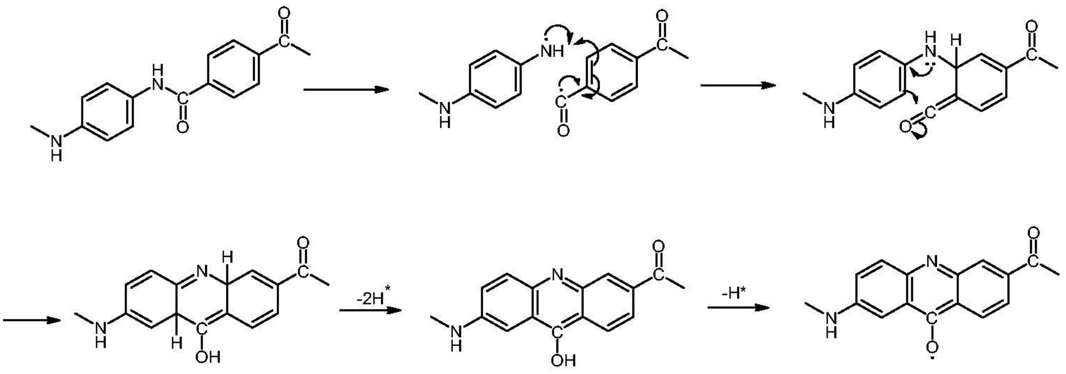
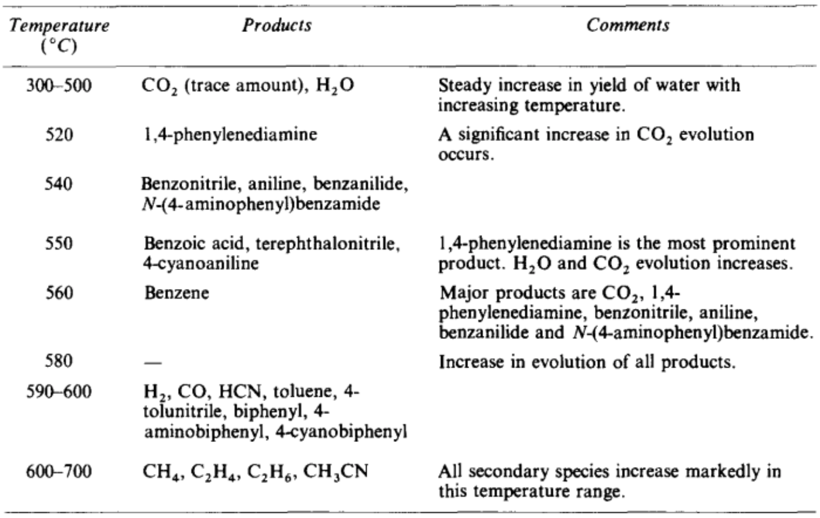


Fig. 5. Speculated thermal decomposition mechanism of Kevlar [53].

Water molecule for the hydrolysis reaction was expected to come from the dehydration of ammonium end groups generated by the interaction of terminal amino groups. Homolytic bond cleavage was more common at high temperatures, and heterolytic bond cleavage was more common at lower temperatures [66], [67]. However, the homolytic cleavage of the HN-CO bonds and aromatic -NH bonds are considered to be the initial reaction of the degradation process [68]. These are followed by -NH protonation, dehydration of intermediate end groups, and heterolytic cleavage of the NH-CO bond, all of which have been validated by other studies. [33], [66], [69].

Table 1. Temperature-dependent pyrolysis products of Kevlar 49 [66].



Temperature (°C)	*Products*	*Comments*
300–500	CO_2 (trace amount), H_2O	Steady increase in yield of water with increasing temperature.
520	1,4-phenylenediamine	A significant increase in CO_2 evolution occurs.
540	Benzonitrile, aniline, benzanilide, *N*-(4-aminophenyl)benzamide	
550	Benzoic acid, terephthalonitrile, 4-cyanoaniline	1,4-phenylenediamine is the most prominent product. H_2O and CO_2 evolution increases.
560	Benzene	Major products are CO_2, 1,4-phenylenediamine, benzonitrile, aniline, benzanilide and *N*-(4-aminophenyl)benzamide.
580	—	Increase in evolution of all products.
590–600	H_2, CO, HCN, toluene, 4-tolunitrile, biphenyl, 4-aminobiphenyl, 4-cyanobiphenyl	
600–700	CH_4, C_2H_4, C_2H_6, CH_3CN	All secondary species increase markedly in this temperature range.

As shown in Table 1, below 500 °C only water and trace amounts of carbon dioxide were detected. At 520°C, 1,4-phenylenediamine was the initial structurally related product evolved. Thereafter, the formation of hydrolysis products, followed by nitriles and compounds containing phenyl end groups, followed by the formation of biphenyl derivatives, was observed for Kevlar, in the temperature range from 300 to 700 °C and many degradation products such as benzene, benzanilide, amino-benzanilides, cyano-benzanilides, and cyano-amino-benzanilides are commonly observed [66], [70]. The evidence of nitrogen oxides being formed during the thermal decomposition of Kevlar was also observed [71].

3.3.Investigation of Kevlar thermal stability

To study the thermal stability, the evolution of the weight with respect to heating rate, the degradation temperature, and the behaviour at specific temperatures, Thermogravimetric analysis performed on Kevlar at a heating rate of 10 °C/min under nitrogen showed that about 55 wt% of the material is lost from 550 °C to 650 °C and further slower degradation from 560 °C onwards [72]. The initial phase of the decomposition started at 526 °C [53] at a heating rate of 10 °C/min in air, and at 20 °C/min initial decomposition occurred at 548.1°C [73]. Also, at a heating rate of 10 °C/min, in argon, maximum mass loss initiates lower than 570 °C.

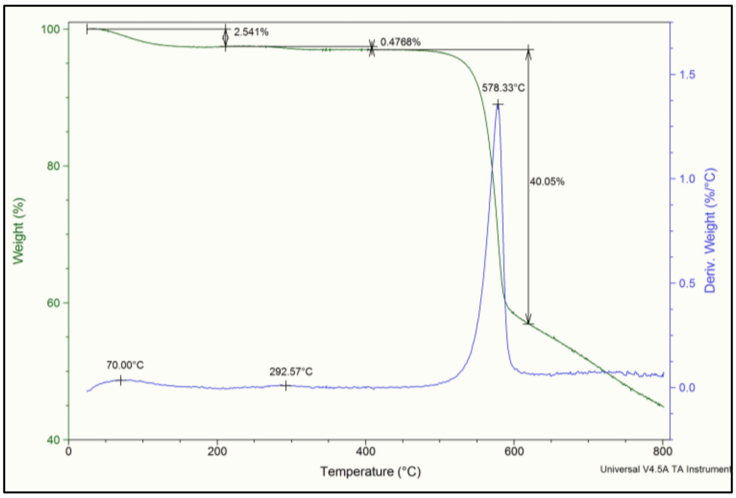


Fig. 6. Thermal degradation of Kevlar fabric by TGA. (Reevaluated based on [64], [72], [73])

The TGA curve (shown in Fig .8) indicates the thermal decomposition of Kevlar at a heating rate of 20°C/min, under N_2 flow, by demonstrating the weight loss (%) as a function of temperature increase; when the temperature rises, weight percentage drops as a result of various reactions that occur. When the weight percentage changes, the first stage of decomposition in the TGA curve begins at a lower temperature and continues all the way to a high temperature. The final segment of the curve denotes the material crystallization, and the temperature at which crystallization begins varies depending on the composition of the material. Nonetheless, TGA alone does not give any information about the thermal decomposition products that are released as volatiles. For this reason, TGA is often coupled with other analytical techniques such as mass spectrometry (MS) or Fourier-transform infrared spectrometry (FTIR) [74].

3.4.Heating and Atmospheric conditions

When exposed to argon, Kevlar thermally degrades in a single step, with a mass loss peak occurring at 579 °C and a corresponding mass loss of 39.74%. When the temperature reaches 1000 °C, there is a 59.73% mass loss. In air, a high mass loss peak is seen for Kevlar at 570.2 °C and a lesser one at 694.2 °C, which equates to mass losses of 39.4% and 92.72%, respectively. At 1000 °C, Kevlar has a mass loss of 98.42%, in air [71]. Contrary to air, under N_2 atmosphere, in the temperature range of 300 to 400 °C, there was a slight weight loss (0.3-0.5% of starting weight), which may have been caused by concurrent oxidation. Above 500°C, the (exothermic) weight loss in air grew quickly, reaching a residual weight of approximately 5% at 590 °C. In an environment containing N_2, no appreciable weight loss occurred below 500 °C, and up to 590 °C, rapid weight loss occurred. The subsequent weight loss is gradual;

depending on the heating rate, the total nitrogen weight loss at 700°C is between 40 and 50% [64].

3.4.1. Influence of natural gas (biomass generated)

Recent developments have shown that the utilization of biomass in the pyrolysis process serves as an additional source of heat, lowering the amount of energy needed for the process and enhancing the quality of the end products. The term "biomass resources" refers to a broad category of sources, including agricultural, wood (including solid briquette), or forest remains [75]. Charcoal is a type of solid biomass fuel that is derived from the process of carbonization, also known as pyrolysis, of wood. It is claimed that charcoal generates a more efficient and sustained fire, with higher temperatures and longer burning times, compared to coal. Although carbon monoxide can be produced during the incomplete combustion of coal, it is generated at high levels when charcoal is burned [76]. Karthik et.al, utilized charcoal, being inexpensive and readily available, as a source of CO_2, as well as heat to facilitate the pyrolysis process of Kevlar wastes, which contributes to lower production costs [46].

3.4.2. Influence of CO2 gas (from salts)

CO_2 has been used as a reaction medium in pyrolysis in order to increase efficiency. The use of CO_2 in the pyrolysis of biomass feedstocks has been experimentally proven effective in increasing pyrolysis thermal efficiency [77]–[79]. It improves the efficiency of thermal cracking of volatile species generated during pyrolysis and may alter product yield (i.e., the composition of the gas, liquid, and solid phases in the pyrolytic product) and product characteristics [80]–[82]. CO_2 can increase pyrolysis of biomass and volatile condensation, while also encouraging tar cracking and inhibiting tar polymerization [83], [84]. When biomass is pyrolyzed in CO_2, it decomposes more deeply than when it is pyrolyzed in N_2 [85].

In order to prevent any oxidation in the pyrolyzing atmosphere, it has recently been revealed that ammonium bicarbonate can be used as a cheaper substitute and a source of CO_2 production during the pyrolysis of Kevlar [86]. Ammonium bicarbonate ($(NH_4)HCO_3$), an inorganic compound, decomposes above 36°C, releasing gases such as ammonia and carbon dioxide; it can also be broken down into these gases by strong acids and bases described as follows [87]: NH_4HCO_3 $NH_3 + H_2O + CO_2$. Ammonium bicarbonate does not produce an alkaline sodium carbonate residue when it is heated to decomposition, in contrast to sodium bicarbonate. Therefore, it doesn't affect the pH of the decomposed product [88].

4. Summary of the results achieved and new findings

4.1.Thermal degradation of Kevlar and its volatile compounds

Understanding the thermal decomposition of materials holds significant importance in relation to their structural and thermal characteristics. In addition, it has been observed that the resultant product from the breakdown of a material can have significant implications in the context of high-temperature scenarios, as evidenced by various studies [68], [69], [82]–[84]. Consequently, it is crucial to examine the thermal decomposition mechanism of a polymer material. The production of carbonaceous materials is subject to various factors, including the type of precursor utilized, the utilization of activating agents, and notably, the pyrolysis process conditions [11], [12], [33].

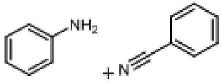
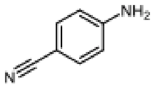
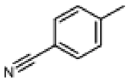
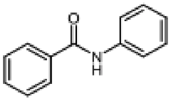
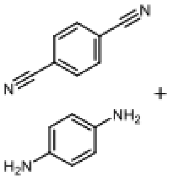
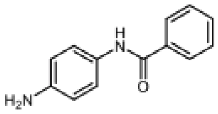
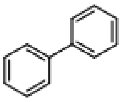
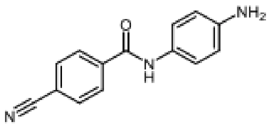
Thermal decomposition can produce carbonaceous (char) or inorganic residues (originating from heteroatoms included in the original polymer, either within the structure or as a result of additive inclusion), or a combination of the two [89]. According to previous research, a process of fiber dehydration is conducted to remove moisture and volatile components from the raw material. Subsequently, pyrolysis and carbonization are employed in an inert atmosphere to generate heat, which facilitates the disintegration of molecular bonds and the formation of carbon structures, through non-isotropic and non-graphitizable structures [35], [61]–[64], [90]. There are different commercially available techniques often selected to identify degradation products of materials. However, the pyrolysis of polymeric waste materials, as a scheme of upcycling, and subsequently acquiring significant information on its thermal degradation process and volatiles thereof, demands simple, unsophisticated, and economic approaches.

4.1.1. Analysis of volatile products of Kevlar pyrolysis

Indirect information has usually come from the characterization of the pyrolyzate, and thus numerous studies on the pyrolysis of Kevlar and other aramid polymers have dealt with the analysis of compounds that evolved during pyrolysis. There are several analytical techniques that have been explored, in order to gain more information on the thermal degradation of Kevlar and the volatile products exposed therefrom. The different techniques that are often chosen to identify degradation products majorly include mass spectrometry, Fourier-transform infrared spectrometry [53]; Infrared spectroscopy [10], [69]; pyrolysis-gas-chromatography/mass spectrometry (Py-GC/MS) [53], [65]; pyrolysis-field ionization mass spectrometry (Py-FIMS) and pyrolysis-gas chromatography (Py-GC) [70]; Electron paramagnetic resonance (EPR) [71];

The identified products of the pyrolytic decomposition of Kevlar at 600 °C in an inert atmosphere are reported in Table 2 [53].

Table 2. Major products identified by Py-GC/MS of Kevlar at 600 °C in an inert atmosphere [53].

Product	Name	Product	Name
	aniline + cyanobenzene		p-cyanoaniline
	p-tolunitrile		benzanilide
	p-dicyanobenzene + p-phenylenediamin		4'-aminobenzanilide
	phenylbenzene		4-cyano-4'-aminobenzanilide

The experimental procedure in the present work allows for volatile products of pyrolysis to be collected by condensing the fumes on a cold surface (in our case, a glass slide), which is then dissolved in isopropanol (IUPAC name: Propan-2-ol). For 24 hours, the dissolved solutions are kept in a cool, dry environment to guarantee that all of the condensed pyrolysis volatiles have completely dissolved. Utilizing an Ultraviolet–visible **(**UV-Vis) Spectrophotometer and a StellarNet diode array (resolution 2nm), the UV-Vis absorption spectra of the samples in isopropanol, is analyzed, as shown in Fig. 9. This is followed by the potential identification of volatile compounds, by means of a spectral matrix decomposition algorithm to decompose measured spectra into possible spectra of individual components [86].

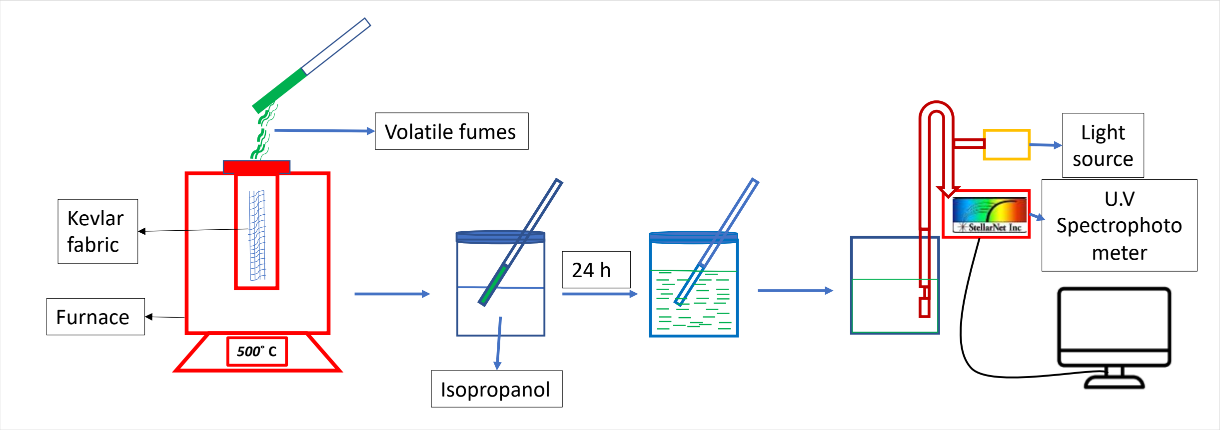


Fig. 7. Schematic illustration of the continuous process of pyrolysis, condensation of volatiles, and subsequent UV-VIS Spectroscopy analysis [91].

4.1.2. Elemental composition of carbonized Kevlar

The significant elemental composition (C, N, O) of the pyrolyzed materials obtained from Kevlar, at various conditions of temperature and atmosphere are shown in Table 3. The experimental evidence reveals that an increase in temperature from 600 °C to 1000 °C resulted in a corresponding increase in the proportion of carbon. The carbon content that was measured exhibits a high level of concurrence with the values that have been previously documented in the literature [69], [92]. A reduction in the content of oxygen and nitrogen has been observed. The observation made suggests that Kevlar macromolecules undergo decomposition at elevated temperatures, leading to the elimination of various constituent elements. The composition of the pyrolyzed material comprises hydrogen and sulfur in low amounts, and a small quantity of other impurities, including S, Na, Cu, Cl, and Ca, which constitute the remaining percentage.

Table 3. Effect of pyrolysis conditions on the elemental composition of Kevlar and carbonized Kevlar.

Treatment conditions	**T** (°C)	**C** (wt %)	**N** (wt %)	**O** (wt %)
Untreated [93], [94]	20	69.07	11.44	14.35
Air [94]	600	68.67	11.58	15.27
N_2 gas [94]	600	69.86	11.71	13.61
	700	68.62	11.47	14.25
Argon [69]	~600	73.2	10.1	11.7
	~1000	75.4	5.5	17.0
CO_2 from charcoal [46]	800	69.17	8.66	13.19
	1000	89.26	2.56	5.87
Mixed gasses from ammonium bicarbonate salt [91]	600	76.1	8.3	12.0
	1000	88.5	1.4	8.3

4.1.3. Ultraviolet-visible (UV–Vis) spectra of volatiles

UV–Vis spectroscopy, applicable to any type of suspension, is one of the most robust and straightforward techniques for quantifying the portion of light lost as it travels through a sample, relative to its blank counterpart [95]. Although UV-Vis spectra are generally insufficient for identifying compounds precisely as compared to Py-GC/MS, It has recently been reported that UV-Vis spectra of a suspension, along with the assistance of an algorithm for the separation of mixture spectra using spectral matrix decomposition, such as singular value decomposition, to decompose measured spectra into potential spectra of individual components, can be used to identify compounds. Following this, the constituent class of volatile chemicals created at the required wavelengths of the two components was retrieved from a database, This offers us an estimated idea of the types or classes of volatiles that are produced throughout the pyrolysis process [86]

In the present work, UV-Vis spectroscopy is used to quantitatively determine elemental concentrations of volatiles collected (as described in section) during the pyrolysis of Kevlar, on the basis of Beer-Lambert law which states that the absorbance of a suspension is directly proportional to the concentration of the absorbent present in the solution and the path length. According to the Beer-Lambert Law, the type and concentration of molecules are significant in the process of radiation absorption [96]. The Beer-Lambert Law is a useful tool for calculating the outcomes of spectroscopic tests (for example, the concentration of an absorbent, the extinction coefficient of the absorbing material, and so on), as in the case of a single compound, which is usually written as:

$$\log_{10}\frac{I_o}{I} = \mathcal{E}\, c\, l \quad \text{or} \quad A = \mathcal{E}\, c\, l \tag{1}$$

where: I_o – intensity of incident beam; I – intensity if transmitted beam; A – absorbance; ε - molar decadic extinction constant; c – concentration of absorbing species; and l – path length [96].

However, for multiple dimensions, it can be expressed as [97]:

$$\mathbf{A} = \mathbf{S}\,\mathbf{C} + \mathbf{E} \tag{2}$$

$$\boldsymbol{A} = \begin{bmatrix} a_{11} & \cdots & a_{1j} & \cdots & a_{1s} \\ \vdots & & \vdots & & \vdots \\ a_{i1} & \cdots & a_{ij} & \cdots & a_{is} \\ \vdots & & \vdots & & \vdots \\ a_{n1} & \cdots & a_{nj} & \cdots & a_{ns} \end{bmatrix} \tag{3}$$

where **A**(n x s) is the matrix of measured absorbances, a_{ij} is the absorbance in j-th sample (spectra) at i-th wavelength, **S**(n x m) is the matrix of absorption coefficients, **C**(m x s) is the concentration matrix, **E**(n x s) is the matrix of errors, n is the number of wavelengths (corresponding to wavelength resolution of the spectrophotometer), s is number of samples (spectra) and m is number of different absorbing compounds present in the samples. Each column of matrix A is in fact one discrete UV -VIS spectrum corresponding to a given sample. It is considered, $\mathbf{A} = \mathbf{U}\mathbf{D}\mathbf{V}^{-1}$ be a singular value decomposition of the spectral absorbance matrix **A** with $\mathbf{U}(n \text{ x } s)$, diagonal $\mathbf{D}(s \text{ x } s)$, $\mathbf{V}(s \text{ x } s)$ with columns of **U** and rows of $\mathbf{V}^{-1}$ sorted in decreasing order of diagonal elements of **D**. **(**containing on diagonal singular values expressing relative importance of individual components of mixture spectrum). Assume that number of real compounds (individual components) is smaller than number of samples and individual concentrations are not in the same ratios. Then the dimensionality can be reduced to assumed k compounds (individual components in mixture spectrum) by decreasing the size of the matrices (cutting off rows/columns) to $\mathbf{U}_k\,(n \text{ x } k)$, diagonal $\mathbf{D}_k\,(k \text{ x } k)$, and $\mathbf{V}_k\,(s \text{ x } k)$, then $\mathbf{A} = \mathbf{U}\mathbf{D}\mathbf{V}^{-1}$ becomes

$$\mathbf{A} = \mathbf{U}_k \mathbf{D}_{\mathrm{k}} \mathbf{V}_k^{-1} + \hat{\mathbf{E}} \tag{4}$$

Where $\hat{\mathbf{E}}(n \times s)$ can be interpreted as the matrix of estimated errors. By introducing a rotation matrix **R** of an appropriate size [98], [99], (4) can be re-written as

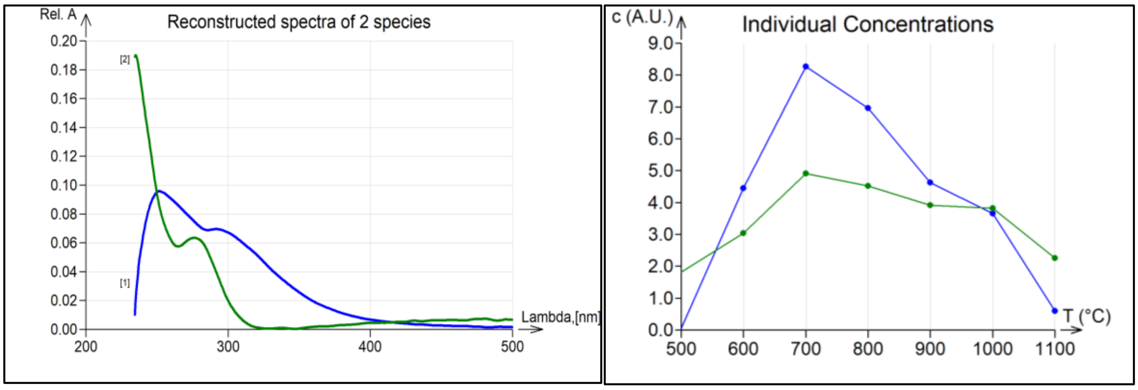
$$\mathbf{A} = \left(\mathbf{U}_k \mathbf{D}_{\mathrm{k}} \mathbf{R}\right)\left(\mathbf{R}^{-1} \mathbf{V}_k^{-1}\right) + \hat{\mathbf{E}} \tag{5}$$

It can be seen that (2) is a special case of (5) implying that the factors in brackets are reconstructed spectra of the assumed individual compounds and their concentrations in samples for a particular **R**.

$$\begin{aligned} \left(\mathbf{U}_k \mathbf{D}_{\mathrm{k}} \mathbf{R}\right) &= \mathbf{S}^* \approx \mathbf{S} \\ \left(\mathbf{R}^{-1} \mathbf{V}_k^{-1}\right) &= \mathbf{C}^* \approx \mathbf{C} \end{aligned} \tag{6}$$

Since both concentrations and absorption coefficients are non-negative numbers, the rotation matrix should ensure that all elements of the two matrices be non-negative. There exist many strategies how to find optimal **R**.

As a criterion the roots for a minimum of selected column of **S**, $\arg\min_{\mathbf{R}} \left\{ \left(S^*_{\cdot j} \right)^2 \right\}$ is used. This algorithm is general based on the Beer-Lambert law validity; this algorithm is oriented to decompose spectra into m components according to criterion the roots for a minimum of selected column of matrix S (eqn. 5). Other programs are obviously decomposing arbitrary spectrum without use the information about their origin (Beer-Lambert law) and number of components is not optimized [65], [100]. Two components were here identified automatically as optimal by program. For two assumed components the reconstructed individual spectra and concentrations according to eqn. (5) are shown in Fig. 10 (a) and (b).



(a) (b)

Fig. 8. Reconstructed UV-VIS spectra (a) of the two assumed components, and (b) of the two identified components at temperatures 500 – 1100°C [86].

Typically, the reconstructed spectra of the assumed components (in the present work, two components) may potentially comprise a mixture of compounds. The chemical identification of pyrolysis products has been previously documented through a variety of methods [1, 6, 7, 9, 10, 12, 15] (also illustrated extensively in section,). Czégény and Blazsó [101] reported that the decomposition pyrograms of Kevlar, as analyzed by Py-GC-MS, yielded benzonitrile, aniline, benzeneamine, and benzoic acid as the primary products in the temperature range of 600-800°C. The formation of benzonitrile was attributed to the homolytic cleavage of the aromatic -NH bond, followed by protonation and dehydration. In addition, a multitude of compounds including benzaldehyde, benzoic acid, benzoate, and aniline were synthesized as reported in reference [28]. This study utilized an 'Organic Compounds Database' to identify the constituent class/types of compounds present at the specified wavelength maxima of the two components. It is noteworthy that certain volatile compounds identified in prior research, fall under one of the categories of volatile compounds obtained in the current study [86]. It is found that one of the components identifies aniline (n,n-dimethylaniline, n-ethylaniline, 3,5-dichloroaniline, p-iodoaniline); and the other component identifies benzonitrile (tolunitrile, chlorobenzonitrile, bromobenzonitrile) which has been confirmed previously [28], [101].

4.2. Development of porous and electrically conductive activated carbon fabric from Kevlar wastes

The majority of textile waste treatment methods do not fully maximize the potential of textile materials and lack economic viability. The conversion of specific textile residues into carbon or partially carbonized structures presents notable economic benefits. The control and optimization of carbonization processes are crucial in the production of carbon materials that exhibit specific characteristics such as tuned porosity, sorption capacity, thermal resistance, and electrical conductivity. These properties are greatly dependent on the precursor utilized [23], [24]. The utilization of Aramid fibers, which are highly ordered polymer precursors, has been studied for the production of activated carbon fiber, and it is noteworthy that these precursors do not require oxidative stabilization, unlike PAN-based precursors [30]. The rigid-rod structure and high degree of aromaticity exhibited by Kevlar make direct carbonization a viable method for obtaining carbon. The fibrous wastes of Kevlar have demonstrated their suitability in the production of porous activated carbon due to their exceptional inherent structure and minimal ash content. This material offers several benefits over other carbon sources, including its cost-effectiveness, high density, superior purity, and lack of contaminants [46]. The conversion of Kevlar woven fabric wastes into activated carbon can be achieved through physical activation by means of a straightforward and regulated thermal treatment process. Optimization of the variables involved in the carbonization process can lead to the attainment of a greater specific surface area and enhanced electrical conductivity of the resulting product. Such carbonized products have the potential to serve as highly effective materials for electromagnetic shielding purposes [46].

4.2.1. Preparation of carbon structures from Kevlar fabric wastes

From the pyrolysis-based upcycling of various polymeric fiber wastes, there are numerous possibilities for the preparation of improved carbon structures, based on the mode of heating and type of heating equipment, heating rate, and internal atmospheric conditions. Carbonized materials obtained from upcycling involve the thermal decomposition of waste polymeric materials in order to recover carbon-rich products. This method has been the subject of substantial research in the field of fiber recovery.

There are various carbonization procedures that can be used, including microwave heating [102], cyclic oxidation [103], and hydrothermal processes [104]. To prepare carbon structures, however, we describe straightforward, cost-effective direct carbonization procedures in the current work. Distinct from our previous work on the development of carbon functional materials from acrylic fibrous wastes, we here demonstrate a novel and direct process of carbonization to produce carbon functional materials from Kevlar woven fabric wastes, without the need for an intermediate stabilization step. This is done at carbonization temperatures varying from 500 – 1200 ºC and without any holding time. Fig. 11. shows the different stages of the production of activated carbon fabric from Kevlar fibrous wastes [46].

The effectiveness of the pyrolysis process, as well as the yield at the end of it, can be improved by using one of several different carbonization atmospheres. Ar_2 (94), N_2 (88), and CO_2 (101-103), are some examples of these. It has been demonstrated through experimentation that including CO_2 increases the thermal efficiency of the pyrolysis process, and deeper decomposition relative to N_2 (109). In the present work, we utilize charcoal [46] and ammonium bicarbonate salt [91] (in separate methods) as sources of producing CO_2 and mixed gasses, respectively, which are inexpensive and commonly available in comparison to N_2 gas. This is done in order to potentially avoid the presence of oxygen inside the furnace during the carbonization process.

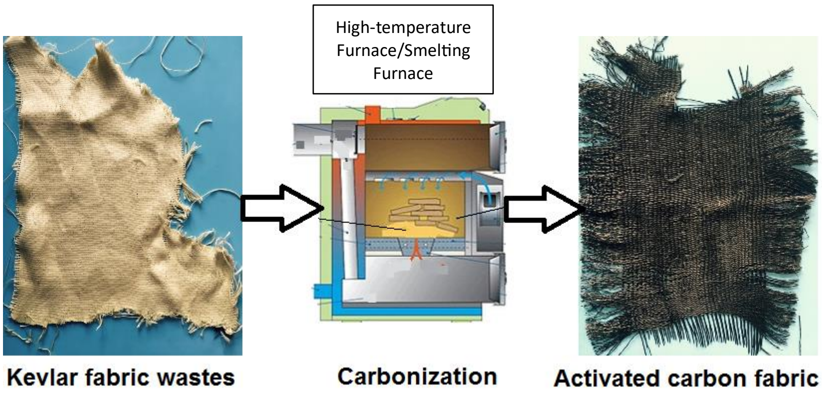


Fig. 9. Schematic of activated carbon fabric preparation from Kevlar fibrous wastes [46].

4.2.2. Yield of carbonized product from Kevlar

The carbon-rich products obtained by various methods are listed in Table 5, with details of their yields in percentages. In all cases, the yield of activated carbon is reduced with an increase in carbonization temperature. The present work involved the implementation of three distinct methods based on atmospheric conditions inside the furnace. Specifically, the present methods utilized the CO_2 produced from charcoal, a combination of gases derived from ammonium bicarbonate salt (NH_4HCO_3), and Nitrogen gas (N_2), respectively. It can be seen that the use of charcoal contributes to higher yield at higher carbonization temperatures, which makes it a remarkable and cost-effective substitute in comparison to yield from other atmospheres. In the presence of charcoal, the yield is about 31% at 1200 °C, which still is substantial, relative to other atmospheres.

Table 4. Yield of Kevlar-derived activated carbon.

Atmospheres	Heating rate (min/°C)	Carbonization temperatures (°C)	Yield (wt%)
Air	10, 10	600, 650	32 [105], 10 [106]
Argon	10, 20	650, 800	65 [105], 41 [107]
Nitrogen	2, 10	650, 650	61 [93], 45 [106]
Charcoal (CO_2)*	5	800, 1000	58, 36
Ammonium bicarbonate salt (CO_2)*	~55	800, 1000	30, 23
Nitrogen*	20	800, 1000	46, 34

* reported in the present work [46], [86], [91]

4.2.3. Physical and morphological characteristics of Kevlar-derived activated carbon

Along with the change in chemical composition, the physical properties undergo modification during the carbonization process, to eventually produce highly porous and light-weight activated carbon structures [32]. The physical features of activated carbon fabrics produced from Kevlar are altered by the carbonization temperature, as shown in Table 6. After carbonization at 1200 °C, it was discovered that the areal density and thickness of Kevlar fabrics decreased by more than 50%. Due to increased porosity, decreased inter-fiber/yarn friction, and abrasion at fiber/yarn cross-over locations brought on by the decomposition of organic materials from Kevlar fabric during the carbonization, it was determined that stiffness decreased with an increase in carbonization temperature. Unlike our prior experience with acrylic fibers [108], the carbonization of the Kevlar fabrics resulted in a small change in their dimensions, which suggested that Kevlar fabrics have superior thermal stability because of the highly ordered arrangement of macromolecules.

It is interesting to note that carbonized Kevlar has been proven to be more flexible than conventional carbon and Kevlar fabric (see Fig. 12). This suggested that the activated carbon textiles being created had better drape qualities, which would increase comfort when used as personal protective structures. In general, although lacking in mechanical strength, the carbonaceous products produced at higher carbonization temperatures preserved their fabric shape and possessed structural integrity, making them appropriate for handling for potential application and additional analysis.

Table 5. Effect of carbonization temperature on physical and mechanical properties of activated carbon fabric produced from Kevlar [46].

Sample	Areal density (g/m^2)	Thickness (mm)	Shrinkage	Flexibility	Dusting	Stiffness (N.m)	Breaking force (N)	Breaking elongation (%)
Kevlar	217.6	1.16	-	-	-	40.92±8.13	126.74±9.07	1.79±0.23
800 °C	124.8	0.61	Good	Average	Good	8.48±1.17	2.7±1.30	1.15±0.49
1000 °C	101.3	0.54	Good	Good	Average	5.38±1.01	2.25±1.06	1.67±0.49
1200 °C	90.7	0.46	Average	Excellent	Poor	4.16±0.93	2.09±1.01	0.94±0.27

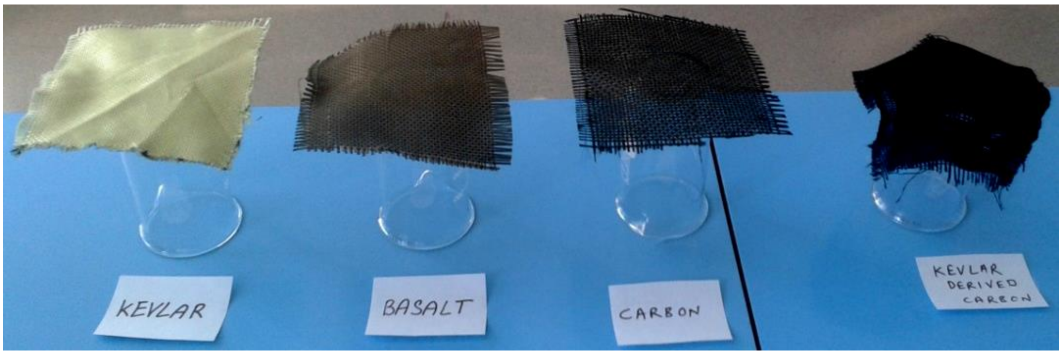
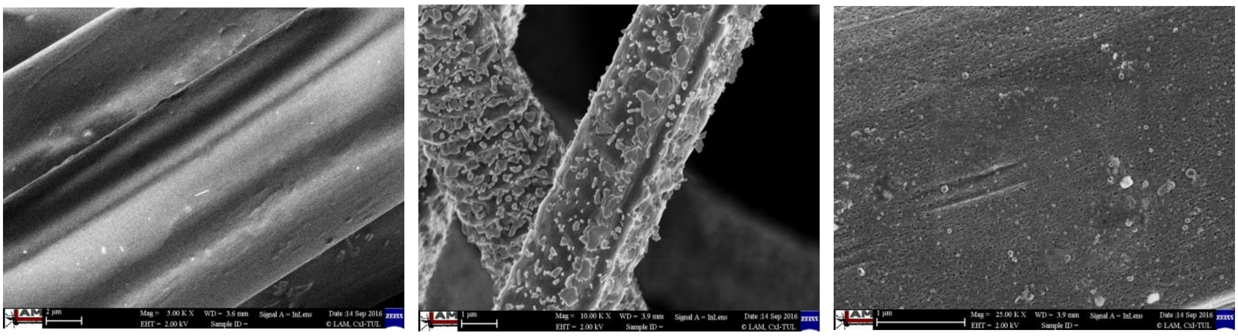


Fig. 10. Visual observation of Flexibility Kevlar-derived activated carbon fabric in comparison to some commercial fabrics [46].

Furthermore, it can be observed that the carbon fibers and activated carbon fibers exhibited a significantly rougher surface in comparison to the unheated Kevlar fibers. This phenomenon was attributed to the generation of additional pores on the Kevlar fibers during the carbonization process [107]. As illustrated in Fig. 3, the surface of the fibers acquired under elevated temperatures exhibited a markedly uneven texture and displayed prominent ridges. Fig. 3(b) depicts the corrugation of carbonized fiber surfaces resulting from the disruption of hydrogen bonds and π–π interactions within Kevlar macromolecules at a temperature of 800 °C. It is observed that the surface roughness exhibited an upward trend in tandem with the rise in the carbonization temperature [34]. This trend was indicative of the emergence of more added pores or rough surfaces subsequent to the physical activation of Kevlar fabric wastes. The emergence of discrete micropores on the surface of Kevlar fibers can additionally be observed upon exposure to a temperature of 1000 °C, as depicted in Fig. 13(c) [108].



(a) Without pyrolysis (b) 800 ºC (c) 1000 ºC

Fig. 11. Microstructure of Kevlar fibers and corresponding carbon fibers under elevated pyrolysis temperatures (a) Kevlar without pyrolysis (b) activated carbon at 800 °C; (c) and activated carbon fabric at 1200 °C [46].

4.2.4. Specific Surface Area of Kevlar-derived activated carbon

Compared to conventional carbon fibers, activated carbon fiber (ACF) has a number of important advantages. High specific surface area, high adsorption capacity, and extremely high rates of gas or liquid phase adsorption are a few of these. It is well-known that due to its highly porous in nature and large pore volume of activated carbon fibers, it has widely been utilized for gas separation, solvent recovery, pollutant removal from water, wastewater treatment, and as a catalyst or catalytic support for various energy storage and conversion processes. Numerous types of materials (both natural and synthetic) have been investigated as precursors since the pore characteristics of activated carbon are mainly dependent on the precursor materials and the synthesis method. Coal, coconut shells, wood, agricultural wastes, or industrial wastes are some of the most often used precursors in the synthesis of activated carbon. For the use of the resulting product, precise structural and surface qualities are crucial, and these features can be described by a variety of physicochemical methods, such as BET (Brunauer-Emmett-Teller) surface area, XRD (X-ray powder diffraction), and SEM (scanning electron microscopy) [109]–[111]. Fig .9 shows the models for pore structure on the basis of the fractal simulation and the displayed model of pores existing in the porous material is expressed using a simulation device consistent with the results obtained from Transmission electron microscope (TEM) observation of a section sample of an ACF [112].

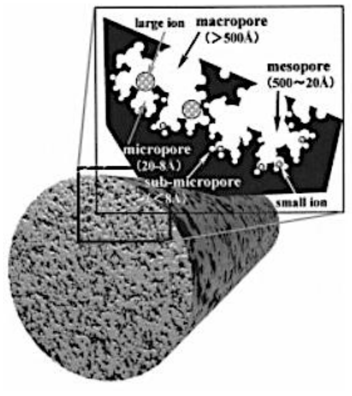


Fig. 12. Porous structure of ACF by fractal simulation.

ACFs with BET surface areas are about 1000 m^2/g are produced when Kevlar is physically activated with CO_2, according to earlier studies in the literature, even at significant burn-offs. Pre-impregnating Kevlar with small amounts of phosphoric acid has a strong effect on pyrolysis and subsequent CO_2 physical activation of the char results in ACFs with surface areas close to 1700 m^2/g and pore volumes that are more than double the values corresponding to ACFs prepared by CO_2 physical activation from non-impregnated Kevlar [32], [33]. It is to be mentioned that similar porous textural characteristics have been obtained with different varieties of Kevlar i.e., either Kevlar pulp or the more crystalline conventional Kevlar [30].

The gasification of non-graphitic carbon and heteroatoms, the reaction with graphitic carbon, and the restructuring of layers of pseudo-graphitic planes are all responsible for the loss of small molecules (CO and CO2) and the creation of improved porosity and surface area during physical activation. An increase in the thickness of the layers of graphitic planes after carbonization and the selective gasification of the less organized carbonaceous material may also be related to the formation of microporosity [88], [113].

In a previous study, by carbonizing Kevlar fibers at 900 °C in an argon atmosphere and then activating them with CO2 at 800 °C, Martinez-Alonso et al. produced ACFs with BET specific surface areas of 986 m^2/g, total pore volumes of 0.50 cm^3/g, micropore volumes of 0.43 cm^3/g, and ultra micropore volumes of 0.28 cm^3/g. Giraldo et al. also synthesized ACFs from Kevlar, but they first carbonized the fibers at temperatures of 800, 900, and 1000 °C under flowing nitrogen before activating them with water vapor at 750 °C for one or two hours. The best material has a micropore volume of 0.24 cm^3/g and a specific surface area of 460 m^2/g. N_2, and CO2, adsorption data indicate that the resulting ACFs are essentially microporous, with a narrow pore size distribution closely associated with the crystalline character of the precursor [72].

The specific surface area of activated carbon fibers plays a crucial role in influencing the electrical properties and electromagnetic shielding behavior through the absorption of the radiations [108]. The porosity and surface area created during the carbonization process is due to the loss of non-graphitic molecules as volatiles and selective gasification of the less ordered fractions of the carbonaceous material, and the reorganization of layers of pseudo-graphitic planes [62]. The specific surface area of carbonized Kevlar fabrics can be obtained by Nitrogen adsorption–desorption isotherm measurements at 77.35 K, using BET (Brunauer-Emmett-Teller) surface area analysis [109]–[111].

The Kevlar chars investigated in earlier studies [30] show a tendency to attain a limit in pore development not previously observed with comparable rayon-based chars [114]. In the latter case, the pore volume per unit mass continues to increase beyond 90% burn-off. An explanation for this behavior can be advanced in terms of the gasification-induced densification effect [30], whereby significant shrinkage of the carbon structure is observed at all stages of burn-off. In the case of carbon dioxide-activated rayon chars, it has been shown that at higher levels of burn-off, both pore widening and pore narrowing can occur [114]. Further, with subsequent activation (physical or chemical) of the carbonized material, the surface area immensely increases with higher micropore volume (≥1000 m^2/g at ~800°C) [34], [69].

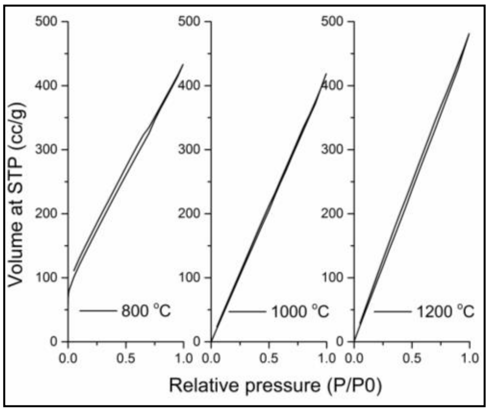


Fig. 13. Adsorption-desorption isotherm of Kevlar-derived activated carbon fabrics [46].

The present work examined the BET analysis of Kevlar-derived carbon fabric structures after carbonization at 800°C, 1000°C, and 1200°C without additional activation. The results showed a type I isotherm from the nitrogen adsorption/desorption isotherm, indicating uniform micropores in the materials (shown in Fig.14.). This classification is consistent with the subdivision proposed by Rouquerol et al [115] and the International Union of Pure and Applied Chemistry (IUPAC) classification [32], [34] [117]. Furthermore, the study revealed the presence of a type IV contribution featuring a type H4 hysteresis loop, a characteristic commonly observed in materials containing slit-shaped pores as described by Gregg et al [116]. Additionally, an increase in carbonization temperature was found to result in a narrower type H4 hysteresis loop, indicating a minimal impact of carbonization temperature on the formation of mesoporosity within the structure [117]. It is observed that there is a small rise in the specific surface area as the carbonization temperature increases. Specifically, the activated carbon fabric that was produced at a temperature of 1200 °C exhibited a specific surface area of 248 m^2/g. The observed specific surface areas, which are relatively lower, and the limited pore development at higher carbonization temperatures, as compared to prior research, may be attributed to the potential pore collapse or closure due to gasification-induced densification, as reported in previous studies [45], [76], [116][113], [118]. Upon a more thorough analysis of the isotherms, it is apparent that the desorption branch exhibits a near-parallel trend in every instance, indicating that the adsorbate has been confined within the highly restricted pore network. At increased levels of burn-off, the low-pressure hysteresis demonstrates a decrease, which suggests that the pore system is becoming increasingly accessible. As a result, a reduced proportion of the adsorbate is confined at low relative pressures [30].

4.2.5. Electrical conductivity of Kevlar-derived activated carbon

Depending on the intended application, the electrical conductivity of activated carbon fibers is a crucial defining property in addition to high porosity. The electrical characteristics of activated carbon fibers are highly variable and mostly dependent on the fiber structure. Higher levels of crystallinity in carbon fibers lead to higher levels of thermal and electrical conductivity. The porous structure is dramatically developed when the carbonized fibers are further activated, which significantly lowers the thermal and electrical characteristics. Additionally, it was discovered that higher degrees of activation remove material from ACF, increasing porosity but decreasing conductivity in the process. The available cross-section for current to travel through the activated carbon fiber diminishes as the surface area increases. Therefore, a decrease in the graphitic organization and the cross-section area accessible for an electric charge to travel through a fiber resulted in a decrease in electrical conductivity. For electrical applications and joule heating, the electrical conductivity of activated carbon fiber is a critical concern [119].

According to one study, the electrical resistivity of activated carbon fibers follows Mott's law with an exponent of 1/2 as illustrated in the equation below [80].

$$\sigma(\mathrm{T}) = \sigma_O \exp\left[-\sqrt{\left(\frac{T_o}{T}\right)}\right] \tag{7}$$

Here, σ_O is the conductivity constant, T is the temperature being measured, and T_o is the fitting parameter that is sensitive to the energy needed for hopping. Exponent 1/3 is preferable to exponent 1/2, according to a different study [120]. By increasing the carbonization temperature, electrical conductivity increases. When ACF is created at higher temperatures, turbostratic structure is more likely to form in the core sections of the fiber, increasing electrical conductivity at higher temperatures. By aligning non-graphitic compounds during the carbonization process, high temperature aids in the production of nanographites with a more ordered structure. Other techniques, like as surface functional group inclusion, heat treatment, and physical adsorption, can also be used to increase the electrical conductivity of ACF [121].

The parallel electrode measurement setup is shown in Fig .15. Here, the sample material is clamped in position along its width by a pair of electrodes at both ends, at a specific distance apart, connected to a multimeter that displays the electrical resistance (in ohms) of the material in series. After which, the electrical resistivity (in ohm. cm) is calculated.

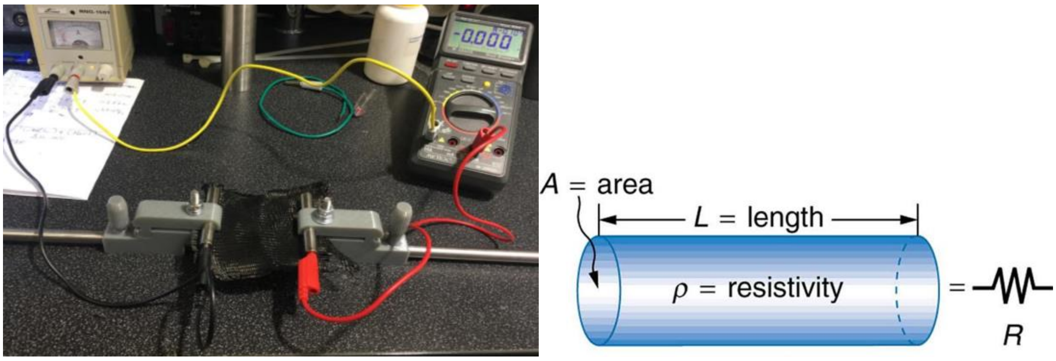


Fig. 14. Parallel electrode measurement setup.

Resistivity is calculated by using the equation,

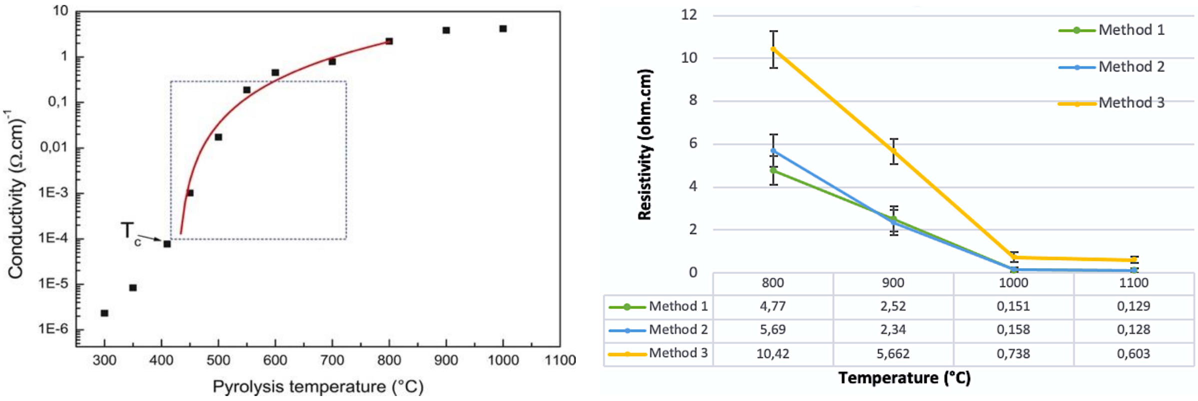
$$\rho = R \; x \; \frac{A}{L} \tag{8}$$

Where, ρ = Surface Resisivity (Ω.m); R = Electrical resistance (Ω); A = Cross-sectional area of the electrode (m^2); L = Distance between electrodes (m). It has been investigated that both surface and volume resistivity of Kevlar-derived carbon products decreased with increased carbonization temperature, using the concentric electrode method [46]. In another study, the electrical resistivity of the Kevlar fabrics was carbonized to four temperatures ranging from

800–1100 °C, using the parallel electrode measurement setup. It is reported that the electrical resistivity decreased progressively with increasing carbonization temperatures, which is attributed to the higher carbon yield due to carbonization at higher temperatures due to the removal of organic compounds in the form of volatiles [91]**.** The movement of electrons through each graphite layer or the hopping of electrons across the defects/interfaces between disordered graphite layers can be attributed to the development of electrical conductivity in activated carbon samples [28], [122], [123].

The electrical conductivity inherent to carbon materials is primarily determined by the voids present within their constituent particles and grains. The correlation between the total pore volume of the AC sample and its intrinsic electrical conductivity is straightforward. Specifically, an increase in the total pore volume of the AC sample is associated with a decrease in its intrinsic electrical conductivity [124]. The graphitization degree of AC materials is a significant factor that strongly influences their electrical conductivity magnitude, as widely acknowledged. According to reports, there is a progressive decrease in electrical resistivity as the carbonization temperature increases. This phenomenon is considered to be instigated by the higher carbon yield resulting from carbonization at higher temperatures, which leads to the removal of organic compounds in the form of volatiles [46][123]. The impact of pyrolysis temperature on the DC electrical conductivity of a range of AC pellets [125]. The results indicate a significant rise in conductivity as the carbonization temperatures were increased within the range of 410 to 600 °C, as illustrated in Fig. 16(a).

It has been previously reported, the resistivity in unidirectional carbon fiber reinforced polymer composites along the fibers is observed to be low, measuring 0.022 mΩ m, while the resistivity transverse to the fiber direction is comparatively high, measuring 310 mΩ m [126]. The resistivity of carbon fibers varies between 2 and 20 μΩ m, contingent upon the specific type of fiber [127]. According to Zhao et al [128], the incorporation of approximately 1.0 wt% CNTs resulted in an increase in the electrical resistivity of CNT/polymer composites to approximately 0.1 Ω.cm. This study involves the preparation of activated carbon fabrics derived from Kevlar through single-step thermal treatments using three distinct atmospheres. These atmospheres include CO_2 evolved from charcoal (method 1), a mixture of gases from ammonium bicarbonate salt (NH_4HCO_3) (method 2), and Nitrogen gas (N_2) (method 3). The electrical properties exhibited by these activated carbon fabrics are noteworthy (as shown in Fig. 16(b)) [91].



(a) (b)

Fig. 15. (a) The direct current, room temperature conductivity versus pyrolysis temperature [125]; and (b) Electrical resistivity of activated carbon fabrics from methods 1, 2, and 3[91].

The optimization of pyrolysis conditions can be tailored to meet specific electrical property requirements. The present work observed a significant decrease in surface and volume resistivity of approximately $15x10^3$ and $25x10^2$ times, respectively, for the activated carbon sample activated at 1200 ºC compared to the sample activated at 800 ºC. The enhanced electrical conductivity of the activated carbon sample at 1200 ºC, as depicted in Table 7, can be attributed to the increased graphitization process. The observed impact of temperature could potentially be attributed to the graphitic structure, as higher temperatures can facilitate the development of such a structure and consequently enhance conductivity [129], [130].

Table 6. Effect of carbonization temperature on the electrical conductivity of activated carbon fabric [46], [91].

<table>
<tr><td colspan="4">Concentric Electrode Method - Surface resistivity (ohm)</td></tr>
<tr><td>800 ºC</td><td colspan="2">1000 ºC</td><td>1200 ºC</td></tr>
<tr><td>1.60 x 10^6</td><td colspan="2">486.15</td><td>95.78</td></tr>
<tr><td colspan="4">Concentric Electrode Method - Volume resistivity (ohm.cm)</td></tr>
<tr><td>800 ºC</td><td colspan="2">1000 ºC</td><td>1200 ºC</td></tr>
<tr><td>967.14x 10^3</td><td colspan="2">1251.29</td><td>414.14</td></tr>
<tr><td colspan="4">Parallel Electrode Method (ohm.cm)</td></tr>
<tr><td>800 ºC</td><td>900 ºC</td><td>1000 ºC</td><td>1100 ºC</td></tr>
<tr><td>4.77</td><td>2.52</td><td>0.151</td><td>0.129</td></tr>
</table>

The phenomenon of electron tunneling, wherein electrons are capable of traversing the interstitial spaces between closely situated grains and particles, has been widely acknowledged as the principal mode of electrical conduction in carbon-based granular and powder materials [131]. The reduction of inter-particle distances and the consequent increase in inter-grain contact are expected to significantly augment the electrical conductivity of the specimen [132]. In addition, the electrical properties are affected by the oxygen-free Lewis-basic sites present on the graphene sheets that are produced through the heat treatment of AC samples. The hypothesis suggests that the emergence of operative groups and configurations on the surface of activated carbon is due to the interaction between oxygen, which has a high electronegativity, and carbon atoms that donate electrons, which are situated on the periphery of the nanographene sheets. This interaction leads to a specific localization of the conduction electrons, resulting in an elevation of the electrical resistance [131]–[133].

4.3.Electromagnetic Shielding Effectiveness of Kevlar-derived activated carbon

Although there have been numerous research studies on electromagnetic interference (EMI) shielding materials in the past, the creation of new, lightweight shielding materials with strong absorption and weak secondary reflection has become necessary for improvements in EMI shielding effectiveness that are more environmentally friendly. Although research has focused on the porous morphology, large specific surface area, and increased electrical conductivity of shielding materials as critical variables, there is still a significant challenge in creating lightweight structures with outstanding EMI shielding qualities on an economic basis [134]–[138].

Absorption, reflection, and multiple internal reflections are the three mechanisms contributing to the overall efficiency of electromagnetic radiation or EMI shielding. Typically, reflection serves as the primary EMI shielding technique. A carrier of unpaired electrons or holes, which interact with the electromagnetic field in the radiation, is required for a material to reflect EMI. Absorption, a secondary EMI shielding mechanism, is influenced by the material's thickness. With electric and/or magnetic dipoles in the shield that can interact with the EM radiation, shielding through absorption becomes more effective. Multiple internal reflections, also known as EM radiation reflection occurring from numerous internal surfaces, phase interfaces, and inhomogeneities in the shielding material, is the third EMI shielding method. [57], [139]–[141]. Only the regions near the surface of the electric conductors can be penetrated by high-frequency electromagnetic radiation, known as the ‘skin effect.’ As the electromagnetic wave travels through the conductor, its strength exponentially decreases [142]. The skin depth (δ) is the depth at which the electric field decreases to 1/e (e is Euler's number, and 1/e is equivalent to 0.37 of the incident value) and is represented as:

$$\delta = \frac{1}{\sqrt{\pi f \sigma \mu}} \tag{9}$$

Where: f – frequency of electromagnetic radiation, σ – shield electric conductivity (Ω^{-1} m^{-1}) a μ – shield magnetic permeability, $\mu = \mu_o\mu_r$, where μ_r = shield relative magnetic permeability, and μ_o – permeability of air or free space ($\mu_o = 4\pi \times 10^{-7}$ H m^{-1}). It is clear that as the EM wave frequency, magnetic permeability, and conductivity of the shield increase, so does the skin depth. The potential interaction between EM waves and shielding materials is depicted in Fig. 19. Absorption loss (SE_A) occurs when the main body of the material absorbs a portion of the electromagnetic waves. Reflection loss (SE_R) results from EM waves being reflected by a material's surface rather than absorbed by it. Multiple reflection loss (SE_M) occurs when absorbed electromagnetic waves travel to a different shielding material interface and are reflected once more. This is followed by energy dissipation inside the shielding material. The three principal types of losses— SE_R, SE_A and SE_M—all work together to attenuate EM waves; therefore, the sum of the three effects above can be used to compute the total SE of EMI (SET), as indicated in the equation below [143], [144]:

$$\mathrm{SE}_T = 10\log_{10}\frac{P_I}{P_T} = 20\log_{10}\frac{E_I}{E_T} = 20\log_{10}\frac{H_I}{H_T} = \mathrm{SE}_R + \mathrm{SE}_A + \mathrm{SE}_M \tag{10}$$

Where P_I, P_T, E_I, E_T, H_I, and H_T stand for the incident power, transmitted power, incident electric field intensity, transmitted electric field intensity, incident magnetic field intensity, and transmitted magnetic field intensity, respectively.

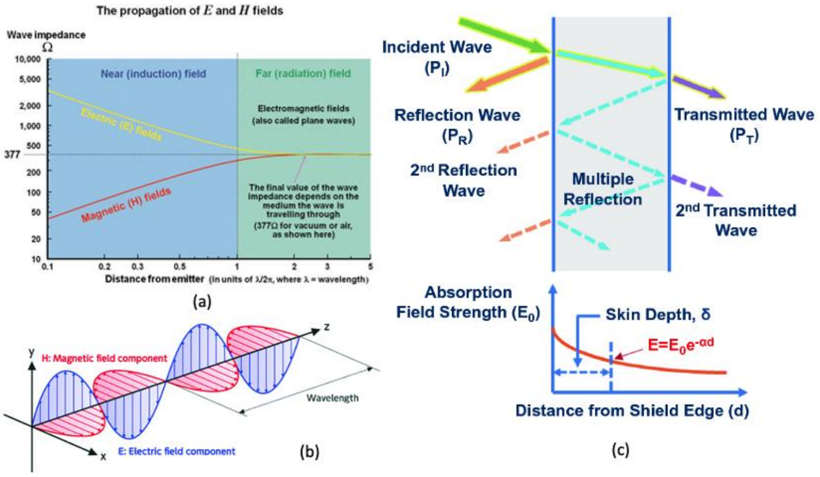


Fig. 16. Schematic representation of the EMI shielding mechanisms (a) Wave impedance and propagation of electric and magnetic fields, (b) electromagnetic transverse oscillating wave, and (c) electromagnetic wave propagation model in EMI shielding materials [142], [143], [145].

The waveguide method, which is used at higher frequencies (e.g., 2.45 GHz), and the coaxial transmission line method, which is used at lower frequencies (e.g., 600 MHz to 1.5 GHz). The waveguide method's measuring setup, which consists of a rectangular hollow waveguide column with electrically conducting walls, is shown in Fig. 20. The test object is positioned at the waveguide column's opening, with a reception antenna inside. A high-frequency analyzer is used to detect electromagnetic signals, and a network analyzer produces electromagnetic waves [108], [134].

According to ASTM D 4935-10, a sample holder with an input and an output that is connected to a network analyzer to produce and receive electromagnetic signals makes up the measuring setup for the coaxial transmission line method, as shown in Fig .21. The fact that the findings obtained in other laboratories are comparable is this technique's main benefit. Additionally, the coaxial transmission line can be utilized to separate each piece of data into its transmitted, reflected, and absorbed components [146].

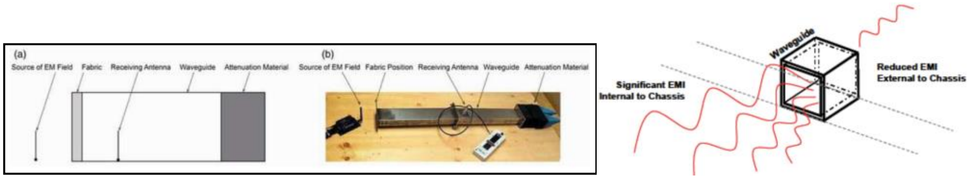


Fig. 17. Measurement setup of waveguide method and its schematic mechanism (bottom) [108], [147].

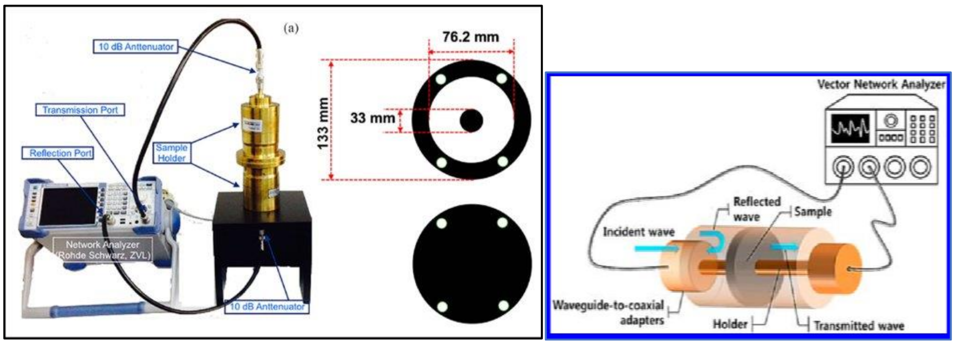
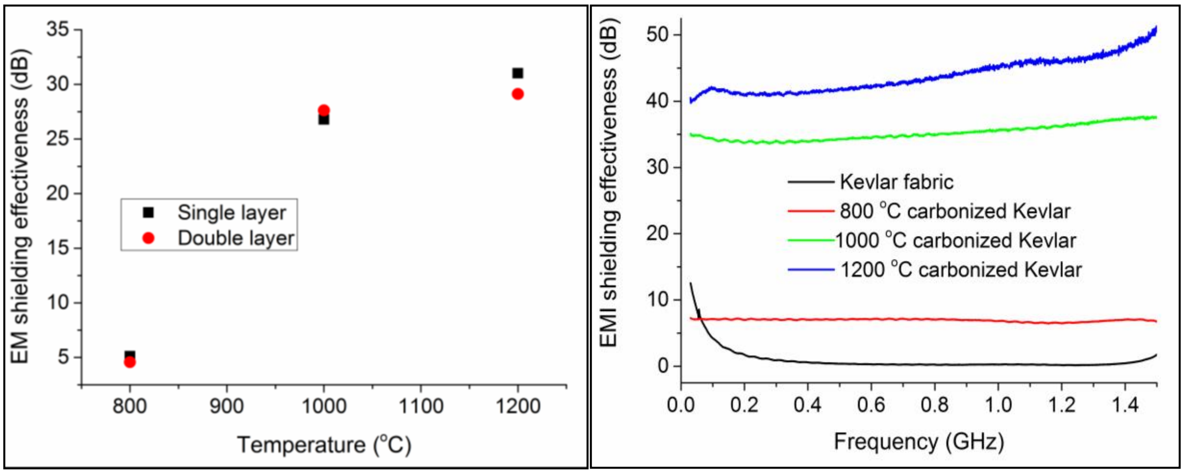


Fig. 18. Measurement set up of coaxial transition line method [137], [148].

The present findings indicate that the activated carbon derived from Kevlar exhibited an increase in electromagnetic shielding efficiency with an increase in carbonization temperature. At a very low carbonization temperature, the shielding efficiency was nearly zero; however, as the carbonization temperature rose, it dramatically increased. A single layer of activated carbon fabric was shown to have EM shielding efficiency of 31 dB, 27 dB, and 5 dB at carbonization temperatures of 1200 °C, 1000 °C, and 800 °C, respectively, at 2.45 GHz frequency as illustrated in Fig. 22 (a). For the most effective shielding efficiency, the percolation threshold is potentially between 1000 °C and 1200 °C carbonization temperatures [46].



(a) (b)

Fig. 19. Electromagnetic shielding effectiveness (a) Effect of carbonization temperature at 2.45 GHz, and (b) Effect of frequency in low-frequency region [46].

Electromagnetic shielding efficiency activated carbon fabrics at different carbonization temperatures in the lower frequency range of 600 MHz, 1 GHz, and 1.5 GHz, is illustrated in Fig. 22 (b). The activated carbon fabric that is produced at a temperature of 800°C exhibited the least effective electromagnetic shielding performance, with a measurement of approximately 5 dB. Upon carbonization of Kevlar fabric at a temperature of 1200 °C, it exhibits shielding capabilities of 42 dB, 45 dB, and 51 dB. The considerably low electrical resistivity of the Kevlar-derived activated carbon fabrics, as discussed in the previous section, enables them to be used as suitable shielding materials Previous studies have indicated that the lower electromagnetic shielding values of activated carbon fabrics derived from Kevlar, in comparison to activated carbon nonwoven web derived from acrylic, can be attributed to both the fiber arrangement and the densification effect induced by gasification [108].

4.4.Joule heating behavior of Kevlar-derived activated carbon fabrics

The principal theory behind Joule heating also referred to as Ohmic heating or resistive heating, is based on Joule's law of conservation of energy, by a phenomenon known as the Joule's effect (shown in Fig.), which is meant to occur when a voltage difference is applied across a conductive material. This effect occurs when electrical energy is consumed in overcoming the resistance in the material between the electrons and the atoms, causing this energy to be generated in the form of heat. Joule's first law postulates that the quantity of thermal energy generated is directly proportional to the square of electrical current, the resistance of the circuit, and the duration for which the current flows through the circuit [149], shown mathematically as follows:

$$Q = I^2 R t \tag{11}$$

where Q is the heat produced by the material; I is the electrical current flowing through the conductor in amperes; R is the electrical resistance in ohms; t is the elapsed time in seconds. The one-dimensional steady-state heat transfer equation is chosen as the model to project the temperature distribution of fibers with applied electric power. The expression for electrical power (Q) produced uniformly by the electrical current is as follows:

$$Q = j \times F = \frac{I^2}{A^2 \sigma} \tag{12}$$

Where F is the electrical field equal to the applied voltage divided by the fiber length, σ is the electrical conductivity of the fibers, and j is current density equal to electric current (I) divided by the effective cross-section area (A). Fig. 23 shows a schematic representation of the Joule heating phenomenon. We assume that heat is transported to the environment at the ends of composite fibers and through the surroundings via air convection in addition to being conducted one-dimensionally through the fibers [150].

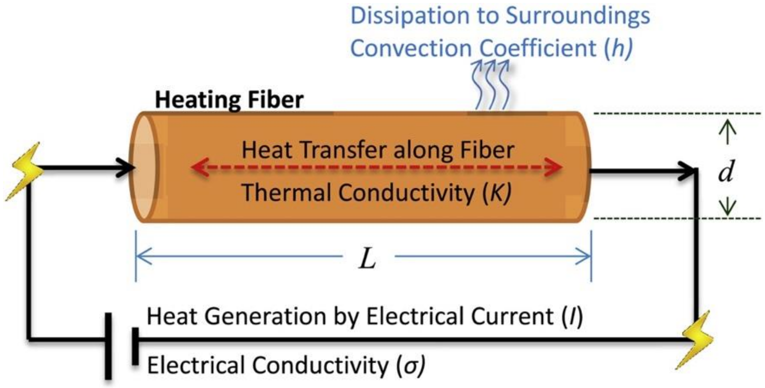
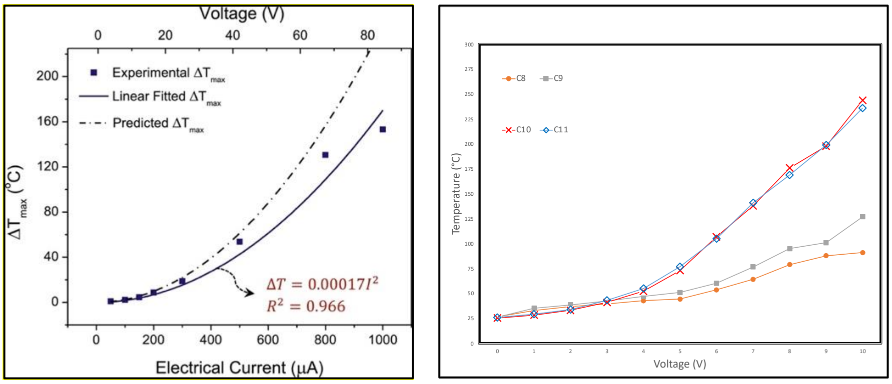


Fig. 20. Schematic diagram of Joule heating phenomenon [150].

In order to produce carbon-rich products suitable for a variety of applications, different polymeric materials have been continuously pyrolyzed and carbonized over time through a process of thermal degradation, where the organic material undergoes aromatic evolution, polymerization, and bond cleavage processes while being subjected to a wide temperature range in an inert environment, the carbonization process removes non-carbon volatiles such as nitrogen, hydrogen, methane, carbon dioxide, carbon monoxide, ammonia, and water, which constitutes about half or more of the precursor's weight. The required type of carbon product and its particular use determine the maximum carbonization temperature, where the chemical composition and the physical characteristics exhibit fundamental changes [38], [69], [80], [151]–[153].

4.4.1. Temperature – Voltage (T-V) relation

It has been reported that the temperature difference is proportional to the electrical power for electrically conductive fibers [150], [154]. Since the electrical power is also proportional to the square of electrical current, the experimental maximum temperature differences obtained by the infrared camera are expected to be linear when fitted as a function of the square of electrical current, as shown in Fig. 24(a), demonstrating that temperature increased with increasing electrical current. At 1000 µA current, the most significant temperature differential recorded for CNT-based conductive fibers was 153 °C [150]. A similar trend is observed in the present work, for fabrics obtained from carbonization of Kevlar (under CO_2 atmosphere from charcoal). From Fig. 24(b) it can be seen that in the temperature-voltage (T-V) curves, the surface temperature remained almost the same, without any significant rise in temperature when a low voltage (V< 3 V) was supplied. Subsequently, the surface temperature of the fabrics increases with increasing voltage. The fabrics carbonized at 1000ºC and 1100 ºC, show a considerably steep increase in surface temperature with increasing voltage. At maximum applied voltage (10 V), surface temperatures of ~250 °C, for fabrics carbonized at 1000°C can be observed [91].

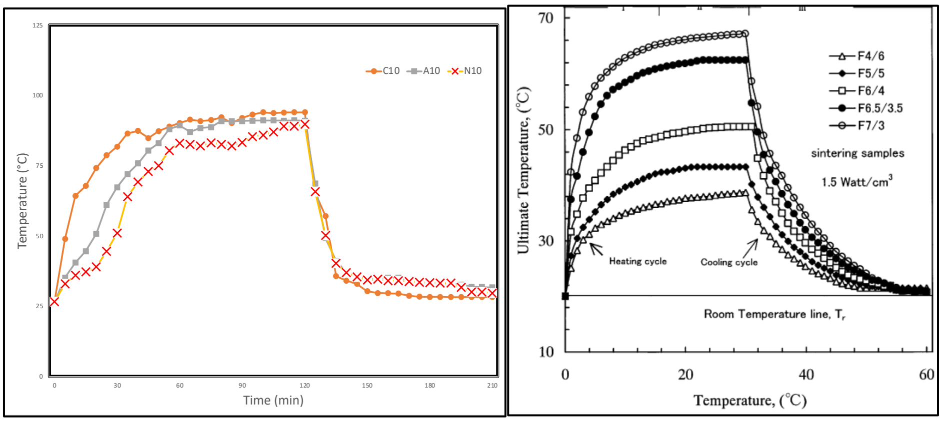


(a) (b)

Fig. 21. Temperature-voltage relation. (a) Maximum temperature difference (ΔT_{max}) estimated for electrically conductive fibers at varying input electrical current and voltage [150], (b) Kevlar-derived activated carbon fabrics [91].

4.4.2. Temperature – Time (T-I) relation: Heating - Cooling Kinetics

The investigation of a heating-cooling cycle through a first-order simple kinetic model can be facilitated by utilizing time-dependent temperature relation curves [91], [155]. The system is comprised of three distinct parameters, specifically the characteristic growth time constant (τ_g), the efficiency of heat transfer (h_{r+c}), and the characteristic decay time constant (τ_d). Fig. 25(a) depicts the time-dependent temperature fluctuations observed in the carbon fabrics derived from Kevlar, which shows a similar trend as compared to time-dependent temperature characteristics of structures containing carbon black at an applied power of 1.5 W/cm^3 (Fig. 25(b)). The data indicate that the surface temperature exhibited a sharp initial increase under a consistent voltage supply of 5 V. Subsequently, the rate of temperature increases gradually decreased over time and ultimately peaked at the 120-second mark. Subsequently, the temperature exhibited a rapid decline towards ambient conditions, reaching room temperature at the 240-second mark, coinciding with the cessation of the applied voltage. The correlation between the steady rise in temperature due to Joule heating and the ultimate carbonization temperature is worth mentioning. Fabrics that have undergone carbonization at temperatures of 1000°C and 1100 °C exhibit a higher leveling-off temperature [91]. Upon conducting additional calculations on the aforementioned parameters, it becomes evident that the values of τ_g and τ_d exhibit a decrease, whereas h_{r+c} displays an increase as the carbonization temperature is raised, due to higher carbon content [155].



(a) (b)

Fig. 22. Temperature – time relation of (a) Kevlar-derived activated carbon fabrics [91], (b) composites with carbon black [155].

5. Conclusion

The present work establishes a concentrated study on the thermal degradation of Kevlar and its volatile products of pyrolysis, and it describes the novelty of the utilization of Kevlar fabric wastes for the development of micro-porous and electrically conductive carbon-based materials by means of a novel, controlled and single-stage processes of carbonization and physical activation, without the requirement of any intermediate stabilization process. The present work focused on the influence of different carbonization temperatures, ranging from 500 to 1200 ◦C, and different atmospheric conditions on the properties of the activated carbon. Here, three different inert atmospheres, namely CO_2 evolved from charcoal, a mixture of gases from ammonium bicarbonate salt (NH_4HCO_3), and Nitrogen gas (N_2), inside a high-temperature furnace were utilized. The following conclusions have been drawn.

- The utilization of Kevlar wastes sourced from industrial processes effectively eliminates the expenses associated with acquiring raw materials. The single-step carbonization process is characterized by reduced time consumption and lower power requirements when compared to other carbonization methods that rely on further activation processes. This feature enhances the importance of upcycling. Moreover, the implementation of charcoal or salts as the heat medium, pyrolysis, and CO_2 atmosphere to augment decomposition, results in a reduction of expenses due to its cost-effectiveness and easy accessibility. These factors significantly impact the expenses associated with manufacturing and production, while simultaneously aligning with the fundamental goal of promoting textile reuse, recycling, and waste management.
- A readily accessible, efficient, and cost-effective experimental arrangement is employed to obtain a preliminary indication of the classes of volatile compounds. This is achieved by utilizing UV-Vis spectroscopy measurements in conjunction with a spectral matrix decomposition algorithm to break down measured spectra into potential spectra of individual components. This algorithm illustrates the progressive changes in concentrations of thermal decomposition products as a function of pyrolysis temperature, as well as their differences in volatility.
- The present work investigated the physical, morphological, and electrical characteristics of Kevlar-derived activated carbon, which was conducted using various techniques such

as EDX, SEM, BET analysis, and electrical conductivity measurements. The yield of activated carbon fabric was determined to be 31% at 1200 ◦C carbonization temperature. Although the mechanical strength of activated carbon fabric greatly declined, it is observed the material retains structural integrity after carbonization. The results indicate a significant reduction in the electrical resistivity of the activated carbon fabrics for all methods of processing as the carbonization temperature increased. Upon subjecting the material to 1200 ◦C carbonization temperature, the activated carbon exhibited a significant decrease in both surface and volume resistivity, of nearly 10^4 and 10^3 times, respectively, compared to the activated carbon sample treated at 800 ◦C, attributed to the presence of a greater amount of graphite, a more uniform distribution of graphite layers.

- The investigation of electromagnetic interference (EMI) shielding efficiency in the high-frequency (2.45 GHz) and low-frequency (1.5 GHz) ranges indicates significant shielding efficiency, attributed to enhanced multiple internal reflections and increased absorption of electromagnetic radiations, consequent to an increased quantity of nomadic charges (most notably, graphite content), uniform dispersion of graphite layers, decreased fiber diameter, an extended mean free path of electrons, larger surface area, higher porosity, and improved conductive network formation in activated carbon subjected to a temperature of 1200 ◦C.
- Furthermore, activated carbon fabrics were found to exhibit a remarkable joule heating phenomenon, which was dependent on both the applied voltage and time. The results indicate that an increase in voltage ($V > 3$ V) led to a corresponding increase in the surface temperature of the fabrics. Surface temperatures of activated carbon fabrics were determined to be 244 ◦C and 264 ◦C at 10 V, attributed to their higher electrical conductivity. The increase in surface temperature through Joule heating in fabrics produced by all three methods was significantly contingent upon the ultimate carbonization temperature. The temperature at which the leveling-off phenomenon occurred was observed to be higher for fabrics that were carbonized at higher temperatures (>1000 ◦C), at a constant voltage of 5 V. It can be suggested that techniques employing charcoal and ammonium bicarbonate salt exhibited superior electrical and joule heating properties, in addition to being economically viable and environmentally sustainable.

6. Future prospects

- To prepare a wide range of particles from the Kevlar-derived activated carbon fibers by ball-milling method.
- To explore the possibilities of modification of particles for selected applications
- To select proper attachments of carbon particles onto textile structures
- To investigate ohmic heating processes and their connections with carbon materials prepared from various polymeric wastes under different conditions.
- Preparation of textile waste-derived porous carbon materials by molten salt-assisted carbonization (Sodium and Ammonium salts) using pyrolysis methods.

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8. List of Publications

Journal publications

[1] **Daniel Karthik**, Karel Kupka, Jiri Militky, Mohanapriya Venkataraman, Study on Thermal Degradation of poly (1,4 phenylene terephthalamide) and its volatile products by unadorned setup of Pyrolysis and subsequent UV-VIS spectroscopy, Journal of Analytical and Applied Pyrolysis, 2023, ISSN 0165-2370, https://doi.org/10.1016/j.jaap.2023.105985 **(IF: 6.437, Quartile: Q1).**

[2] **Daniel Karthik**, Vijay Baheti, Jiri Militky, Muhammad Salman Naeem, Veronika Tunakova, and Azam Ali. 2021. "Activated Carbon Derived from Carbonization of Kevlar Waste Materials: A Novel Single Stage Method" Materials 14, no. 21: 6433. https://doi.org/10.3390/ma14216433 **(IF: 3.748, Quartile: Q2).**

[3] Salman Naeem, Vijay Baheti, Veronika Tunakova, Jiri Militky, **Daniel Karthik**, Blanka Tomkova, Development of porous and electrically conductive activated carbon web for effective EMI shielding applications, Carbon, Volume 111, 2017, Pages 439-

447, ISSN 0008-6223, https://doi.org/10.1016/j.carbon.2016.10.026 **(IF: 11.307, Quartile: Q1).**

[4] Mohanapriya Venkataraman, Jiří Militký, Alžbeta Samková, **Daniel Karthik**, Dana Křemenáková, and Michal Petru. 2022. "Hybrid Prepreg Tapes for Composite Manufacturing: A Case Study", Materials 15, no. 2: 619, https://doi.org/10.3390/ma15020619 **(IF: 3.748, Quartile: Q2).**

[5] **Daniel Karthik**, Jiri Militky, Yuanfeng Wang, and Mohanapriya Venkataraman. 2023. "Joule Heating of Carbon-Based Materials Obtained by Carbonization of *Para*-Aramid Fabrics" C 9, no. 1: 23. ISSN 2311-5629, https://doi.org/10.3390/c9010023 (**Web of Science (ESCI), tracked for impact factor, projected quartile is Q2).**

[6] Palanisamy, S., Tunakova, V., Wang, Y.F., **Karthik, D.**, Militký, J., 2022. EMI Shielding of the Copper/Nickel-Coated Polyester Nonwoven. Solid State Phenomena. https://doi.org/10.4028/p-om1446. **(IF: 0.53, Quartile: Q3).**

[7] **Karthik D.**, Baheti V, Tunakova V. and Militky J.: Carbonization Of Kevlar Fabrics For Effective Emi Shielding Applications, Fibres and Textiles (4), Vlákna a textil, 2018; 41-44. ISSN 2585-8890.

Conference publications

[1] **Karthik, D.**, Militky, J., Venkataraman, M. Eradicating spread of virus by using activated carbon (2020) Textile Bioengineering and Informatics Symposium Proceedings 2020 - 13th Textile Bioengineering and Informatics Symposium, TBIS 2020, pp. 56-63.

[2] **Daniel Karthik**, Vijaykumar Baheti, Jiri Militky, Carbon and Silicon Based Electrically Conductive Elastomeric Textile Sensors, 46th Textile Research Symposium, The Textile Machinery Society of Japan, 2018.

[3] **Daniel Karthik**, Vijay Baheti, Veronika Tunakova, Jiri Militky, Sundaramurthy Palanisamy, Development of Electrically Conductive Activated Carbon Fabric fromKevlar Fabric for Effective Emi Shielding Applications, Textile Bioengineering and Informatics Symposium, Wuhan, China 2017; 1044; ISSN:19423438. DOI: 10.3993/tbis2017.

[4] **Daniel Karthik**, Vijay Baheti, Jiri Militky, Sundaramurthy Palanisamy, Studies on Organic and Inorganic Micro/Nano Particle Reinforced Epoxy Composites, Textile Bioengineering and Informatics Symposium Proceedings, Manchester, UK, 2018, pp.149, ISSN: 1942-3438, DOI: 10.3993/tbis2018.

[5] **Karthik, D.**, Baheti, V., Novotna, J., Samkova, A., Pulicek, R., Venkataraman, M., Srb, P., Voleska, K., Wang, Y., Militky, J. Effect of particulate fillers on creep behaviour of epoxy composites. Materials today: Proceedings. 1. ed. Oxford: Elsevier, 2020Pp. 1–4. ISSN 2214-7853.

[6] Sundaramoorthy Palanisamy, Veronika Tunakova, Benny Malengier, **Daniel Karthik**, Lieva Van Langenhova, Jiri Militky, Development Of The Force Sensitive Resistors

Using Polypyrrole Coated Cotton Woven Fabric For Pressure Sensing Application, 22nd International Conference, STRUTEX, 2018.

[7] **Daniel Karthik**, Vijaykumar Baheti, Jiri Militky CEC 2017, TUL, Liberec, Czech Republic – “Carbonization of Kevlar Fabric for Effective Emi Shielding Applications”

[8] **Daniel Karthik**, A.I. Wasif, Vijaykumar Baheti, Jiri Militky Presented a poster entitled “Study on Gauze Bandages” at STRUTEX conference, December 2016, Czech Republic.

[9] Dana Křemenáková, Dan Wang, Shi Hu, **Daniel Karthik**. Effect of anti-wash treatment on the related comfort properties of the EMI shielding material, Proceedings of the CLOTECH 2022 conference, 2022.

[10] S. Palanisamy, V. Tunakova, Y. Wang, **D. Karthik**, J. Militky, Emi Shielding Of The Copper/ Nickel Coated Nonwoven. Autex Conference 2021.

[11] Palanisamy, S., Tunakova, V., Venkataraman, M., **Karthik, D**., Wiener, J., Militky, J. Modeling of an electrically conductive nonwoven strip for electromagnetic shielding, Textile Bioengineering and Informatics Symposium Proceedings 2021 - 14th Textile Bioengineering and Informatics Symposium, TBIS 2021, 2021, pp. 37–43

[12] Baheti, V., **Karthik, D**., Novotna, J., Samkova, A., Pulicek, R., Venkataraman, M., Srb, P., Voleska, K., Militky, J. Creep Behavior Of Particle Filled Epoxy Composites. The Twenty-Sixth Annual International Conference on Composites/Nano Engineering, 2018. Not numbered (2 pages).

[13] Jiří Militký; Vijay Baheti, Wang Yuanfeng & **Daniel Karthik**, Utilization Of Fibrous Waste For The Preparation Of Advanced Carbon Materials, Book of Proceedings (International textile, clothing & design conference), 2022. ISSN 1847-7275, p.41.

[14] Azam Ali, Vijaykumar Baheti, Jiří Militký, Abdul Jabbar, Hafiz Faisal Siddique, **Daniel Karthik**, Sundaramoorthy Palanisamy, Effect of jute fibre treatment on moisture regain and mechanical performance of composite materials. IOP Conference Series: Materials Science and Engineering, 2017, DOI: 10.1088/1757899X/254/4/042001

[15] Smita Shamsunder Baheti, Maroš Tunák, **Daniel Karthik**, Structure Analysis of Carbonized Needle-punched Nonwovens by X-ray Computed Tomography, STRUTEX – Structure and Structural Mechanics of Textile Fabrics - Conference Book, 2018, ISBN: 978-80-7494-430-7

[16] Yuanfeng Wang, **Daniel Karthik**, Kai Yang, Tao Yang, Xiaoman Xiong, Vijaykumar Baheti, Jiří Militký, Electrical Heating Properties of Carbon Fabric/Green Epoxy Composites Filled with Fly Ash, 47th Textile Research Symposium, 2019, ISBN: 978-80-7494-473-4

Book Chapters

[1] **Daniel Karthik**, Jiri Militky, Mohanapriya Venkataraman (2021). Eradicating spread of Virus by using Activated Carbon. Textiles and Their Use in Microbial Protection: Focus on COVID-19 and Other Viruses.Technical Fibres. 2. ed. United Kingdom: Woodhead publishing (Elsevier), 2018. Pp. 367–419. ISBN 978-0-08-101272-7.

[2] Vijaykumar Baheti, Promoda Kumar Behera, **Daniel Karthik**, Jiří Militký, Fly Ash Based Geopolymer Concrete Materials, Recent Developments in Fibrous Material Science, 403-412, 2015, ISBN: 978-80-87269-45-9.

[3] Promoda Kumar Behera, **Daniel Karthik**, Muhammad Salman Naeem, Vijaykumar Baheti, Jiří Militký, Geopolymer/Fly Ash Concrete Composite Materials, 21st Conference STRUTEX, 225-232, 2016, ISBN: 978-80-7494-269-3

[4] Vijaykumar Baheti, Promoda Kumar Behera, **Daniel Karthik**, Jiří Militký, Properties of basalt fibres suitable in concrete composite, Advances in fibrous material science, 95-118, 2016, ISBN: 978-80-87269-48-0

9. Curriculum Vitae

DANIEL KARTHIK, M.Tech.
17. Listopadu 587/8
Liberec 460 15,
Czech Republic.
danielkarthiktex10@gmail.com
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Education

Ph. D., Material Engineering- Technical University of Liberec (2015- Present)
Thesis: Carbon-based Functional Structures from Pyrolysis of Kevlar Fabric Wastes.
Post Graduate Degree (M.Tech), Technical Textiles - Department of Textile Technology, Shivaji University, Maharashtra, India. (2014)
Graduation (B.Tech), Textile Technology - Department of Textile Technology, VTU, Belgaum, Karnataka, India. (2012)

Research Projects

- Advanced structures for thermal insulation in extreme conditions Registration number: 21-32510M, Grantové agentury České republiky (GAČR)
- Project Ministry of Education, Youth and Sports in the frames of support for researcher mobility, Material Engineering, Faculty of Textile Engineering, Technical University of Liberec, Czech Republic)

Research Internships

- 2016 – Institute: Department of Textile Engineering, Indian Institute of Science, New Delhi, India. (Period: 2 months)
- 2017 – Institute: School of Material Science & Engineering, Georgia Institute of Technology, Atlanta, GA, USA. (Period: 2 months)
- 2018 – Institute: Carbon NEXUS laboratory, Deakin University, Geelong, Australia. (Period: 2 months)

Industrial Collaborations

- Utilization of Kevlar wastes, from Veba, Czech Republic
- Study of Pyrolysis of Kevlar under various conditions and characterization of thermally decomposed products, UV spectroscopy analysis and mathematical modelling, at TriloByte Statistical Software, s.r.o, Pardubice - Staré Hradiště, Czech Republic

Skills and Training

- A creative thinker of innovation and a passion for learning, teaching, and mentoring
- Extensive research experience using a multidisciplinary approach by applying the principles of basic science and engineering
- Pyrolysis and carbonization process of polymeric fibrous systems, and optimization of process parameters towards the development of porous and conductive carbon-rich products
- Experience in analyzing thermal degradation processes and identification of volatile compounds obtained thereof.
- Strong knowledge of analytical and characterization techniques like UV-Vis spectroscopy, Mass spectroscopy, Electrical resistivity, EMI Shielding, Joule heating, XRD, DSC, DMA, TGA, BET surface area and elemental analysis.
- Lab scale pyrolysis methods, firefighting training, First-aid, Basic Lab Safety.
- Excellent computer, oral, written communication, and interpersonal skills
- Eager to optimize global resources, develop novel and sustainable materials, generate significant cost savings, and promote innovative solutions in complex environments.

Awards

- ‘Outstanding Researcher Awards’, Eradicating spread of virus by using activated carbon. In Textile Bioengineering and Informatics Symposium. 2020. (TBIS 2020) Combatting Covid-19 Pandemic with Science and Technology Innovations
- ‘Best paper presentation’, Carbonization of Kevlar Fabric for Effective Emi Shielding Applications, CEC conference, 2017, TUL, Czech Republic.

10. Brief description of the current expertise, research, and scientific activities

Doctoral Studies: Full-time student at the Faculty of Textile Engineering, Department of Material Engineering

Specialization: Textile Technics and Material Engineering

List of Exams Passed:

[1] Heat and Mass Transfer in Porous Media
[2] Structure and Properties of Textile Fibers
[3] Textile Metrology
[4] Mathematical Statistics and Data Analysis
[5] Experimental Technique of Textile

State Doctoral Examination: Passed

11.Recommendation of Supervisor

Recommendation of the supervisor on Ph.D. thesis of Daniel Karthik, M.Tech.

Date: 19.10.2023

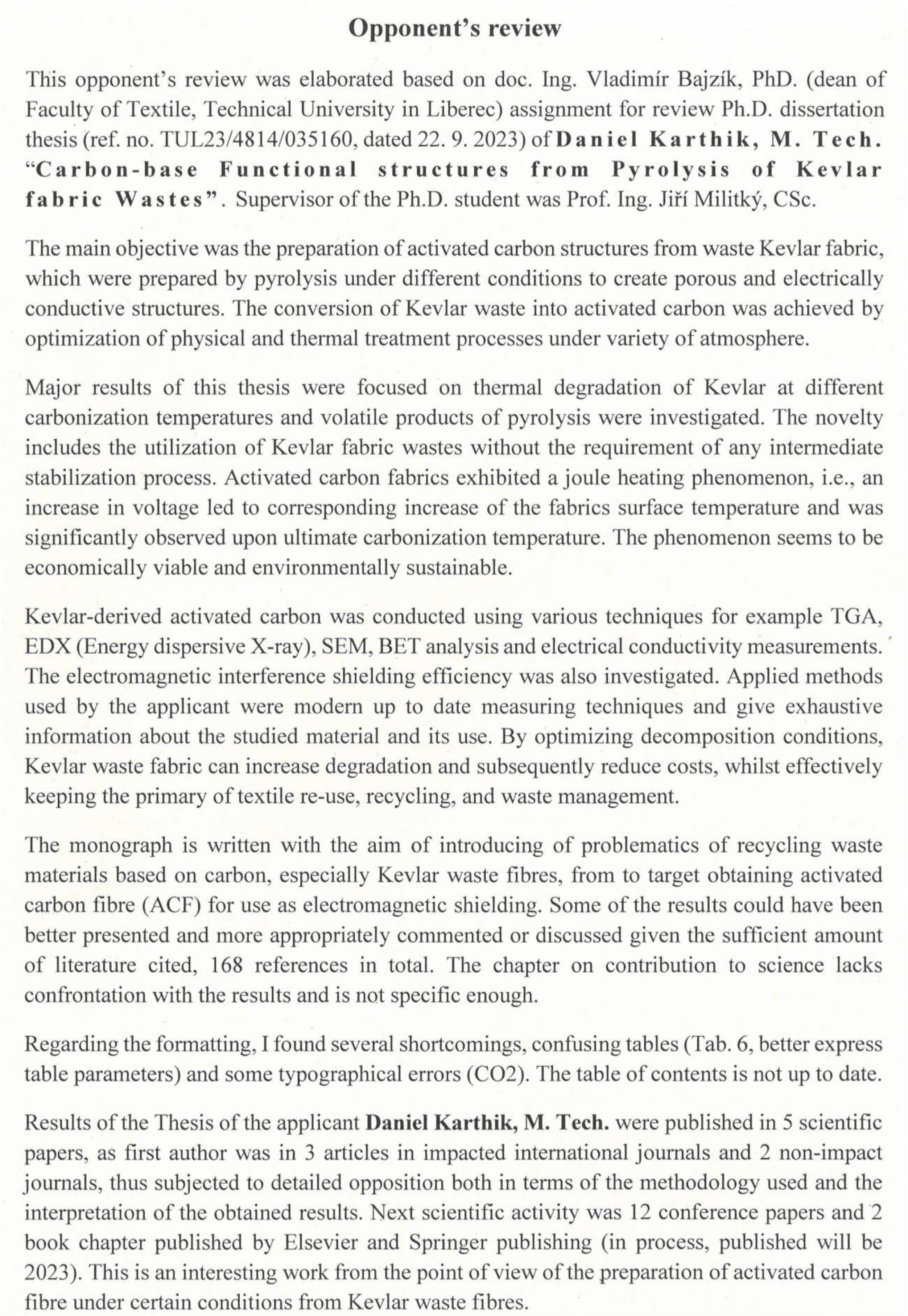
Thesis title: **Carbon-based functional structures from pyrolysis of Kevlar Fabric Wastes**

The PhD thesis of Daniel Karthik is concerned with the utilization of fibrous aromatic polyamide (Kevlar) waste for the development of microporous and electrically conductive activated carbon by means of novel single-stage processes of carbonization. He investigated the thermal degradation of Kevlar and its volatile products of pyrolysis. He studied mainly physical, morphological, electrical conductivity, electromagnetic shielding, and joule heating behavior of carbonized materials under varying process conditions of pyrolysis. He studied the progressive changes in concentrations of volatile products and their differences in volatility from the thermal decomposition of Kevlar depending on the pyrolysis temperature, by demonstrating an algorithm for the separation of mixed spectra obtained by UV spectroscopy.
The thesis adheres to the specified format and successfully achieves all of its intended objectives. The candidate has demonstrated a high level of systematicity over the course of his research, thus achieving outstanding results through the pursuit of specific aims. He employed advanced scientific methods to evaluate and examine data. The discussions pertaining to the outcomes are coherent and include comparisons of the attained results with those of other published works. The language proficiency exhibited in the thesis is commendable and satisfies the standards expected at the doctoral level. Several of his findings exhibit novelty and have already been published in high-impacted academic journals. His exceptional abilities are evidenced by his publication record in journals with high-impact factors. Throughout his research tenure at TUL, he has promoted his findings through the publication of 7 papers in journals with high-impact factors, 4 book chapters, and 17 articles in conference proceedings. Throughout his academic pursuits, he demonstrated a high level of diligence and competency. The findings of the dissertation are valuable, innovative, and readily applicable in practical settings. Thus, it is highly recommended that the thesis be presented for the final doctoral defense.

When searching for plagiarism, a match of 21 percent was found with the habilitation thesis of Ing. Tunáková PhD and match 17 percent with summary of thesis (autorepeat) of Ing. Salman Naeem. In both cases, it is based on joint publication of these workers, which was published in the journal Carbon (2017) and is presented in full both in the dissertation (Karthik) and in the habilitation thesis (Tunáková), which otherwise differ significantly in terms of subject matter. The summary of thesis (Naeem) also contains parts of their joint publication. Therefore, it is not plagiarism, but an incorrect evaluation by the program, which does not consider the option that each of the co-authors chooses the form of a dissertation or habilitation thesis as an annotated overview of published articles, the full texts of which are an obligatory part of it.

Prof. Ing. Jiří Militký, CSc. EURING
Supervisor

12.Reviews of the Opponents



Opponent's review

This opponent's review was elaborated based on doc. Ing. Vladimír Bajzík, PhD. (dean of Faculty of Textile, Technical University in Liberec) assignment for review Ph.D. dissertation thesis (ref. no. TUL23/4814/035160, dated 22. 9. 2023) of **Daniel Karthik, M. Tech.** **"Carbon-base Functional structures from Pyrolysis of Kevlar fabric Wastes".** Supervisor of the Ph.D. student was Prof. Ing. Jiří Militký, CSc.

The main objective was the preparation of activated carbon structures from waste Kevlar fabric, which were prepared by pyrolysis under different conditions to create porous and electrically conductive structures. The conversion of Kevlar waste into activated carbon was achieved by optimization of physical and thermal treatment processes under variety of atmosphere.

Major results of this thesis were focused on thermal degradation of Kevlar at different carbonization temperatures and volatile products of pyrolysis were investigated. The novelty includes the utilization of Kevlar fabric wastes without the requirement of any intermediate stabilization process. Activated carbon fabrics exhibited a joule heating phenomenon, i.e., an increase in voltage led to corresponding increase of the fabrics surface temperature and was significantly observed upon ultimate carbonization temperature. The phenomenon seems to be economically viable and environmentally sustainable.

Kevlar-derived activated carbon was conducted using various techniques for example TGA, EDX (Energy dispersive X-ray), SEM, BET analysis and electrical conductivity measurements. The electromagnetic interference shielding efficiency was also investigated. Applied methods used by the applicant were modern up to date measuring techniques and give exhaustive information about the studied material and its use. By optimizing decomposition conditions, Kevlar waste fabric can increase degradation and subsequently reduce costs, whilst effectively keeping the primary of textile re-use, recycling, and waste management.

The monograph is written with the aim of introducing of problematics of recycling waste materials based on carbon, especially Kevlar waste fibres, from to target obtaining activated carbon fibre (ACF) for use as electromagnetic shielding. Some of the results could have been better presented and more appropriately commented or discussed given the sufficient amount of literature cited, 168 references in total. The chapter on contribution to science lacks confrontation with the results and is not specific enough.

Regarding the formatting, I found several shortcomings, confusing tables (Tab. 6, better express table parameters) and some typographical errors (CO2). The table of contents is not up to date.

Results of the Thesis of the applicant **Daniel Karthik, M. Tech.** were published in 5 scientific papers, as first author was in 3 articles in impacted international journals and 2 non-impact journals, thus subjected to detailed opposition both in terms of the methodology used and the interpretation of the obtained results. Next scientific activity was 12 conference papers and 2 book chapter published by Elsevier and Springer publishing (in process, published will be 2023). This is an interesting work from the point of view of the preparation of activated carbon fibre under certain conditions from Kevlar waste fibres.

Questions to be answered during thesis defense:

1. Explain the differences between results of UV Vis and Infrared spectra of volatile product of Kevlar pyrolysis.
2. When is shrinkage and flexibility after Kevlar pyrolysis good or poor (better is higher or lower).

Based on the latter mentioned facts and by the course of law (Higher Education Law No. 111/1998. Sb.) §47 I recommend to accept the PhD. dissertation thesis of Daniel Khartik, M. Tech. for defense.

In Zlin, October 13, 2023

Doc. Mgr. Barbora Lapčíková, Ph.D.

Associated professor for materials science and engineering
Tomas Bata University in Zlin

Review of dissertation thesis

Daniel Karthik, M.Tech.:

"CARBON-BASED FUNCTIONAL STRUCTURES FROM PYROLYSIS OF KEVLAR FABRIC WASTES"

The provided dissertation comprises 105 pages, including copies of 4 published articles.

An investigation was carried out to explore the thermal degradation of Kevlar and to analyse the composition of volatile compounds resulting from its pyrolysis. The activated carbon structures underwent scrutiny for their geometric, physical, morphological, electrical, and thermoelectric properties.

Abstract:

Abstract is summarizing the content of the work and is relatively brief. It is not defining the proper background (expressed as key background + purpose of the study), methods, results.

List of abbreviations, and List of symbols is not present in the thesis.

Introduction

The introductory section is competently written, provides key information and ideas related to the process of upcycling fibrous waste through pyrolysis and carbonization.

Objectives

Objectives are too short and not clear.

Extended and clear objectives should be there. Author has not shown strong knowledge to explain the thesis significance, scope and objectives. They are very less and not properly explained. Present objectives look like the objectives of a research article rather than a PhD level thesis.

Literature review

Author showed certain negligence to write this part of the study. There are a lot of errors in this chapter, including formatting. A sufficient number of literature sources is divided into chapters with nearly 150 references. The literature part should explain previous work and the most critical literature findings related to the topic.

Methodology

A novel single-stage method on Activated carbon from Kevlar fibrous waste with carbonization and physical activation is declared. Only one method and one textile waste fiber were selected. Perhaps, the author could check the effect of chemical activation before carbonization.

Results and discussions

The chapter summarizes all results of the thesis. Author summarizes the findings of the previous chapters. Figure formatting is different at different places. Rest of the results are in attached publications.

Conclusions

The author explained major conclusions, but generalization of the results and especially comparing different techniques is missing. I would expect more conclusions from the study – get advantages and drawbacks of different techniques of carbonization, and also the effect on different textile fibrous waste, the techniques used and their detailed comparison from various points of view.
The future work planned here is also missing.

Comments and recommendations

The figures numbers in text and in caption are not matching, even some figure captions are missing in text also.

For example:

2. Upcycling of Polymeric wastes to Carbon-based functional materials
Figure mentioned in text is 2….while given in caption 1

3. Carbon-based functional materials
Figure mentioned in text is 3….while given in caption 2
Figure mentioned in text is 4….while given in caption 3 (page 14)
Figure mentioned in text is 5….while given in caption 4 (page 15)
Figure mentioned in text is 1….while given in caption 5 (page 16)

On the same pattern, the whole thesis should be seriously addressed. I suggest the Committee to consider a request to the author to re-submit the thesis file after correcting the errors.

Formatting errors:

- Spaces between the paragraphs are different at different places.
    - Such as the space between main heading 5 and text (page 19) is different from the space between main heading 6 and its text (page 24).

- The text in some paragraphs is not justified.
    - 3.2.1 Paragraph starts from the ideal features of precursor, is not justified.

- Additionally, dot is absent after the reference number 44.
- Somewhere dots are missing such as after references pyrolysis process
    - [103] (page 28). This mistake is repeated also at other places.
    - Some places have additional dots, such as in section 3.1.
    - (1 dot before reference number and 1 dot after reference number).

- The text labelling on most figures is almost not readable.

- The author has directly copied and pasted the Table 1, Table 2. At least these tables should be redrawn at PhD level thesis.

Technical comments:

- Similar work on the development of activated carbon has already been defended as a PhD thesis in the year 2017 at the same Department. Proof is attached in the present thesis as an attachment - the article on the topic:

"Development of porous and electrically conductive activated carbon web for effective EMI shielding applications".

Candidate has used the same parameters for developing porous and electrically conductive carbon. Hence, it looks like there is not much new in this work which would add substantial contribution in already existing knowledge, except the precursor material.

- The author has not mentioned about pore size and pore size distribution at different carbonization temperatures and their potential role on electrical resistivity of developed activated carbon structures.

- The author used the carbonized woven fabric to check the EMI shielding effectiveness. The author has not checked the effect of cover factor of carbonized fabric (at 800 ºC to 1000ºC and 1200ºC) on the EMI Shielding effectiveness.

- The author of the thesis claimed the dramatic decrease in the electrical resistivity as he increases the carbonization temperature from 800 ºC to 1000ºC and 1200ºC. In fact, if it is due to the increase in graphitization.

Questions for defence:

1. How have you justified that the graphitization is increased with increase of carbonization temperature?
2. Can you please explain the novelty of the carbonization method you used, especially compared to the previous work performed at your Department in 2017.

Conclusion:

The concept of the thesis the candidate choose is to present most significant publications with comprehensive comments. There is no doubt the publications prove appropriate scientific competencies of the author team(s) of the articles. As the candidate is the first author of 3 of 4 publications provided, it can be considered to be sufficient proof of his ability to work as independent researcher. However, the quality of the "comments" part of the thesis is not at corresponding (PhD.) level. Hence,

I recommend the thesis for the defence,

but I leave the clear and critic final decision on the Committee.

Liberec, December 11, 2023 doc. Ing. Stanislav Petrík, CSc.