

## 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 (CO<sub>2</sub>). 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. Barбора Lapčiková, 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 °C to 1000°C and 1200°C) 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 °C to 1000°C and 1200°C. 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 Petřík, CSc.

