Referee´s report on PhD. thesis of

Azam Ali

„ Surface Deposition of Metals on Textile Structures "

Professor Miroslav Černík

The presented thesis consists of 106 pages divided into 7 chapters. The thesis´s main objective was to investigate the preparation, properties and applications of surface-modified textiles with electrical conductivity, EMI shielding, ohmic heating capability, and antibacterial properties. The thesis has standard parts - Introduction, Thesis objectives, Literature review, Methodology, Results and discussions, Conclusions and Future works. In the end, there are References and a List of author´s publications. The author´s list of publications is extensive.

First pages
Before the introduction, there are Contents, List of tables, List of figures, List of abbreviations, and List of symbols. The last two lists are incomplete, and the list of symbols contains only 4 symbols, and even very strange.

Thesis significance, scope and objectives
The author made here an introduction to his work and explained the objectives of the study. The work was split into four major parts – an deposition of Cu particles on plain woven cotton fabrics; an deposition of Ag particles on cotton fabric and knitted fabric; electroless platting on NPs coated woven fabrics; development of tribo-electic generators for energy harvesting (platting of Ag on stretchable knitted fabrics). Unfortunately, these topics look as four various subjects without any unification bridge.

Literature review
There is a sufficient number of literature sources divided into ten parts. Even the whole chapter is well written, some parts and Figures are disputable. Directly, Fig.1 shows a range of electric resistivity for different materials, but the ranges are too broad to me (e.g., metals are always in a range of 10^{-5} or smaller resistivity, not from the value in the presented interval); Fig. 2 shows just basis of Ohm´s law, which is too basic for the PhD. thesis. Part 3.5 (Research on electrically conductive metalized fabric) presents many figures. I think the literature part should explain previous work and the most critical literature findings related to the topic. It should not repeat figures published previously (at least in such quantity).

Methodology
There is a vast variety of methods used for the preparation, characterization and application of the fabrics. Four different materials were prepared – Cu particles on textiles, Ag particles on textiles, Cu plating on coated fabrics and electrode Ag plating. There are other methods like antibacterial testing, heating performance, and durability besides electric and electromagnetic testing. I am not sure if all these methods are essential for the final topic of the thesis.

Results and discussions
The chapter summarizes all results of the thesis. For each conductive structure, several measurement was performed – electric conductivity, SEM analysis, mechanisms of attachment,

electromagnetic shielding, antimicrobial properties, and durability of fabrics. Figure 33 repeats the scheme of Fig.22, and again in the chemical formulae, the student shows an ignorance of basic chemical nomenclature (Cu^{+2}, etc.). His knowledge of chemistry, based on many errors in the text, is too basic.

Conclusions
This chapter summarizes the findings of the previous chapters. The author repeated major conclusions for each material, but generalization of the results and especially comparing different techniques is missing. I expected more conclusions from the study – get advantages and drawbacks of each technique and their detailed comparison from various points of view. The future work planned here is also in terms of individual studies of particular techniques.

Referee remarks, question and conclusions
QUESTIONS
1. Prior deposition of Ag particles, cotton fabric was dipped into NaOH. According to Fig. 23, for this pretreatment Na^+ ions are essential. Why? What were the mechanisms of this step?
2. Antibacterial tests in Fig. 36: Has the student performed the tests? Describe details about the 95% confidence interval. Was is determined from the repetition of the experiments? How many parallel experiments have you carried out? Are the error bars calculated separately for each of the values?
3. Equation (9) and (10): I do not understand these equations? Could you give an example, how equation (9) works? The stability constant equation is also wrong.
4. Cathode reactions mentioned on page 79 considers the complete deprotonation of EDTA. This deprotonation depends on the solution pH. At which conditions, such a situation appears?

Imperfections and recommendations
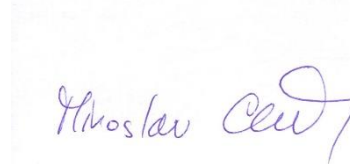The thesis is written in good English but with significant typing errors.
Example on p.13: Micrometric metal are did not cause…; nanoparticles are shown displayed in…; was dip. Chemical formulae are sometimes wrongly written (e.g., p.18, Cu^{+2}, SO_4^{-2}, e for electron w/o the negative charge).

Referee´s conclusion
The presented thesis of Azam Ali has all the formal parts and shows the author is able to carry out scientific work. The thesis shows four different techniques of fabrics´ surface modification, various characterization techniques and potential applications. The work significantly contributes to knowledge in the subject. The work´s fundamental disadvantage is in fragmentation, where the general idea is the application of various techniques, but without their comparison and generalization. The author also showed a problem with the understanding of chemical processes applied in the thesis. The language is acceptable and entirely understandable.

Besides imperfections, errors and weak points, the thesis meets the criteria to be taken to the defense.

In Liberec (Czech R.) on February 14, 2021

Professor Miroslav Černík

Title: *Surface Deposition of Metals on Textile Structures*

Author: Azam Ali, M.Sc.

This presented thesis deals with the current topic of preparation of conductive textiles, which contain applied layers of copper or silver particles and their subsequent use in the field of conductive materials.

Thesis contains 103 pages divided into 8 main chapters and contains 10 tables and 69 pictures.

At the beginning of this thesis, four main goals of experimental work were defined, in-situ deposition of copper or silver on cotton fabric / knitted fabric usable for electromagnetic shielding, electroless plating for further copper deposition and study of silver deposited knitted fabrics as an electrode usable for TrEG self-powered device.

The chapter of literature review of current knowledge in the field of textile materials containing conductive metal layers, their preparation, and various uses, follows. Attention was paid mainly to the issue of electromagnetic shielding and the use of conductive textile materials in medicine. The search is clear and a sufficient amount of resources has been used.

Using methods of preparation and testing conductive materials are sufficiently described for possible repetition. Personally, in order to determine the concentration of metal on the fabric, I would choose not only the SEM EDX, but also the determination of the metal by elemental analysis from the ash of the cotton fabric. On the other hand, this method is destructive for sample.

In the experimental part of present thesis, firstly copper or silver layers were prepared by chemical reduction on the surface of a cotton fabric or knitted fabric. For metal identification appropriate physical methods was used. A positive effect of higher amounts of copper or silver on the determined values of surface resistivity, electromagnetic shielding and antibacterial activity was described.

The effect of extension on the above properties was also observed for the silver coated knitted fabric. The subsequent electroless copper plating over previously deposited copper or silver coated fabrics was the next part of the experimental work, when a significant increasing of conductivity was achieved.

One type of these samples, silver electroplated knitted fabric was used as an electrode for triboelectric generators, where the values of electrical resistance and voltage and current flowing due to the stretching of the knitted fabric were monitored. In the last part of the thesis, presented results were compared with the values published in the literature. This thesis contains the chemical description of individual processes.

Formally, this thesis contains some typing errors, e.g.:

p. 41   … for 12 minutes; … g/L

p. 58   Table 6: Oxygen (O) should probably be the correct element instead of potassium (K)

p. 59   … see 42

I miss of SH and SE in the list of abbreviations. Some citations in the bibliography are not complete, e.g. number 60, 106, 114.

The thesis presents interesting results of experimental work in the field of preparation of textile materials with a conductive layer usable not only as an electromagnetic shield, but also in the field of

medicine. The doctoral student demonstrated good scientific work and published his results sufficiently. He is the co-author of 22 articles in impact journals (11 of them as the main author), 6 chapters in books and presented his results at international conferences.

I also appreciate the proposal for the future direction of further research.

My questions:

1. Why do you choose knitted fabric with Polyamide for Triboelectric generators?
2. Is the difference between deposition of metal particles on cotton and polyamide surface?
3. Did you measure electrical resistivity dependence of number of repeated extensions for silver electroplated knitted fabrics?
4. Did you determine values of durability under washing for higher temperature than 40 degree Celsius?
5. Could you explain increasing standard deviation during increasing Extension presented on Figure 64b, p. 80?

In conclusion, I **recommend** this thesis for the defense.

In Pardubice on July 8, 2020

Ing. Michal Černý, Ph.D.