

Kinetics of Free Radicals Decay Reactions in Cellulosic Based Heritage Materials Disinfected by Gamma Radiation

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INTRODUCTION

Disinfection of cultural heritage artefacts and archived materials by gamma radiation has been successfully applied in recent years. However, more research is still required to study undesirable effects (side-effects) which may appear in sensitive materials as a function of the delivered dose. Conservators and restorers frequently get worried about possible long time effects in irradiated materials (post-effects). During irradiation process, some energetic and unstable chemical species called free radicals appear in the treated matter. They disappear in different ways, interacting each other and some of them interact with the artefact matter being responsible for disinfestation and irradiation side-effects. The kinetics of free radical decay reaction depend among others of the absorbed dose and the properties of irradiated material. In this study, contemporary paper samples were irradiated using gamma radiation from Co-60 with different absorbed doses

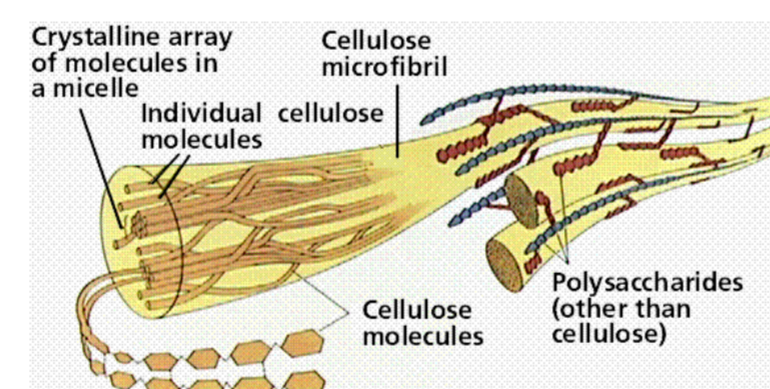
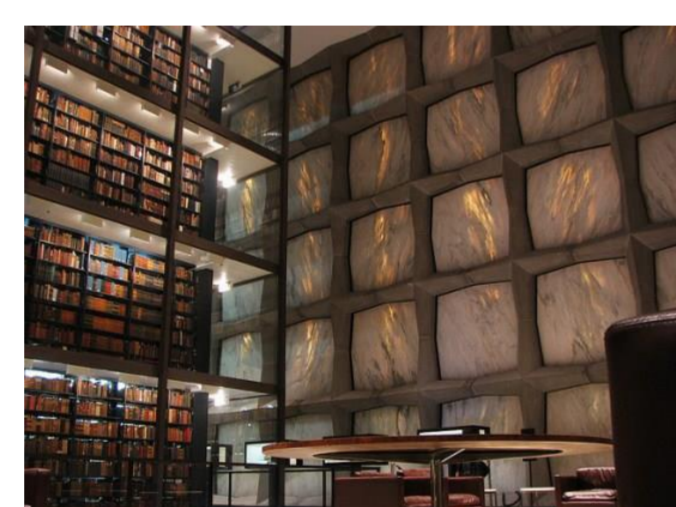
OBJECTIVES

- Study the kinetics decay of the cellulose free radicals induced by irradiation using Electron Paramagnetic Resonance -EPR
- Cellulose structure modifications analyses by ionizing radiation by scanning electron microscopy - SEM/EDS and X-ray diffraction – XRD.

MATERIALS AND METHODS

Contemporary paper samples (2010)

- bleached chemical pulp
- mineral load 10%, pH 4.6
- 100% short fibers



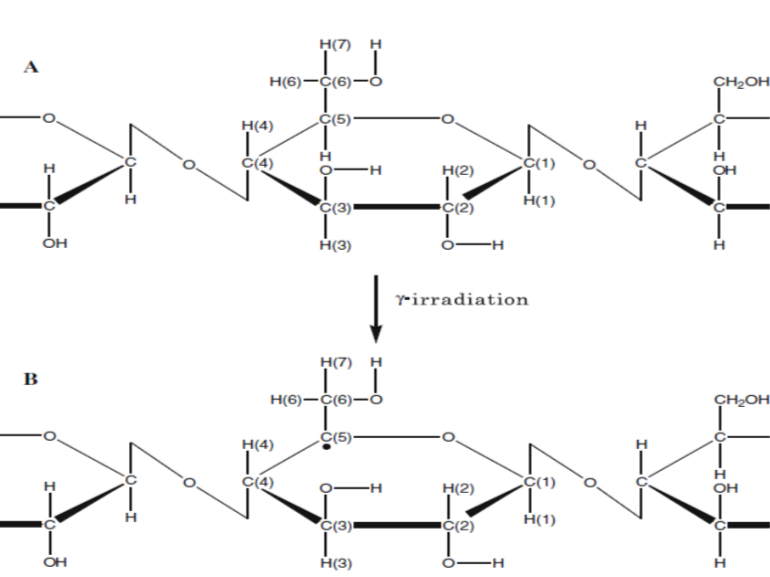
Radiation Processing

Multipurpose Gamma Irradiation Facility –IPEN
Absorbed Doses: 1 kGy – 6 kGy – 10kGy -12kGy -15 kGy
Dose rate: 5 kGy/h



Electron Paramagnetic Resonance

- Time after irradiation measurements: 1.5 to 670h.
- Magnetic field 337.6 to 367.5 mT
- Field modulation amplitude 0.2 mT
- Field modulation frequency 100 kHz
- Microwave power 2 mW.
- Measurements at room temperature, at different times after irradiation.



Material characterization

- Scanning Electron Microscopy(SEM)
- Scanning Electron Microscopy Energy Dispersive Spectrometry(SEM-EDS)
- X-ray diffraction -XRD

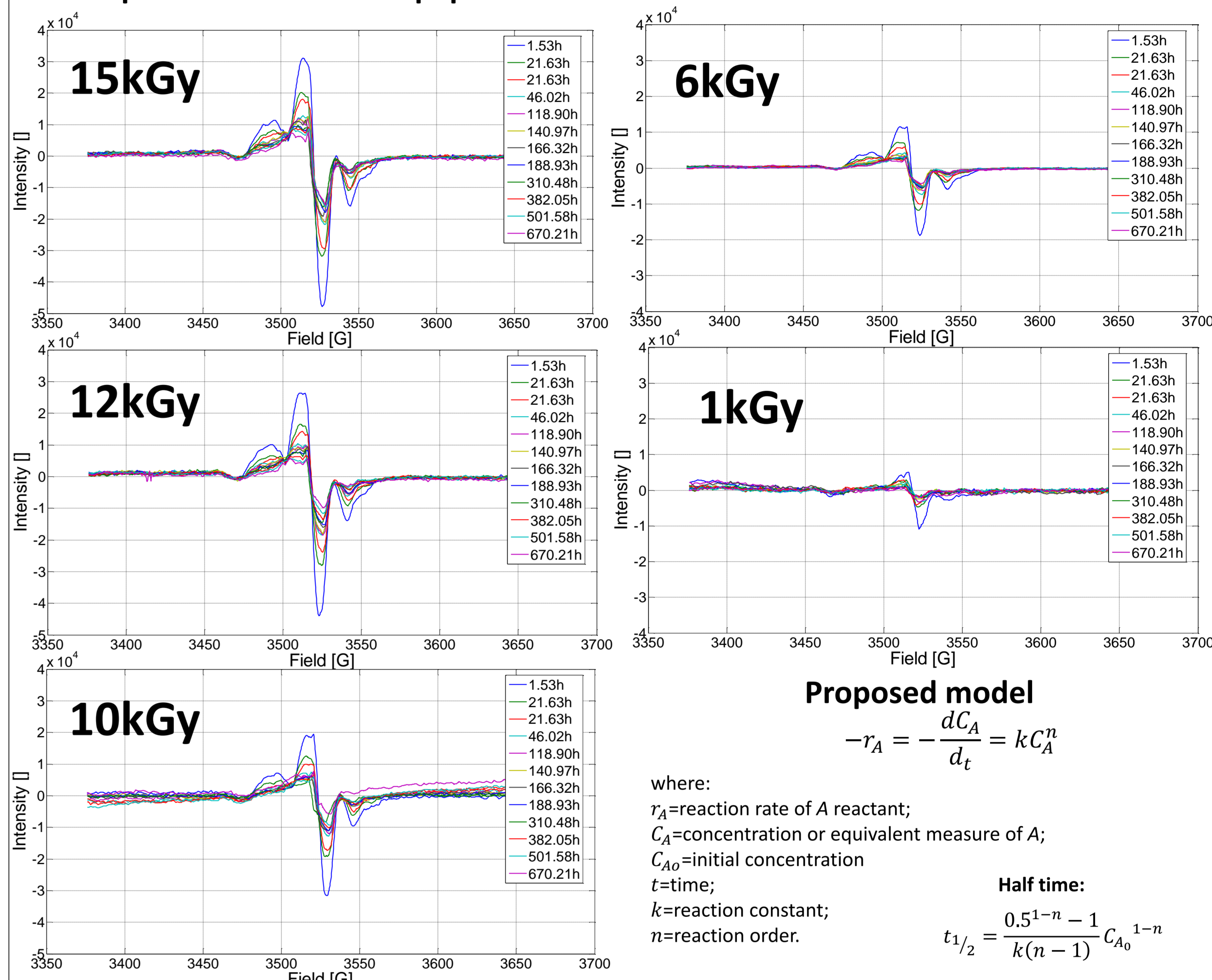


Kinetics models calculations

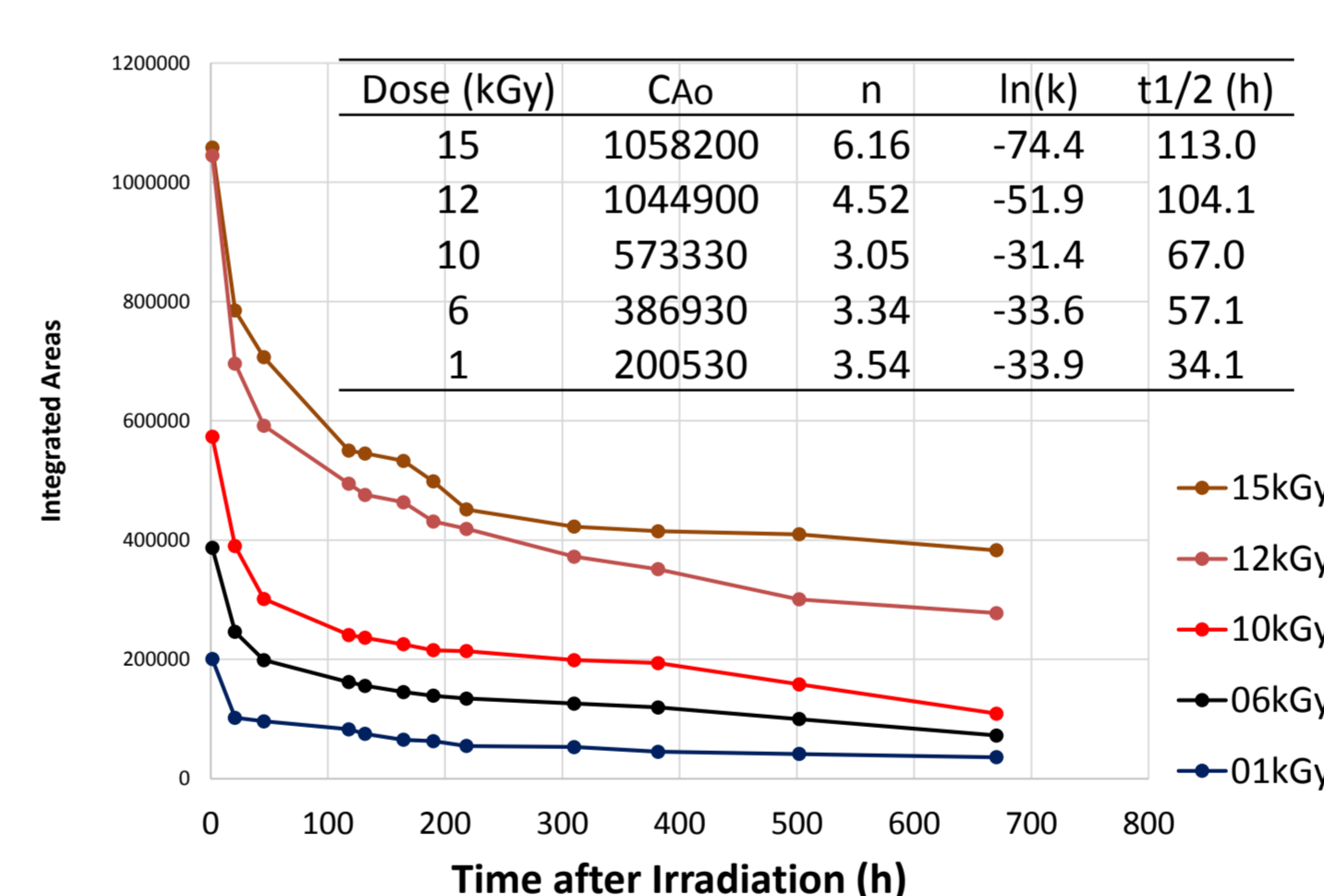
- EPR spectra signals treatment - De-noising by wavelets.
- Integrated total area calculations (CA)
- Order reaction determination by differential method
- Half-life cellulose free radicals determination

RESULTS

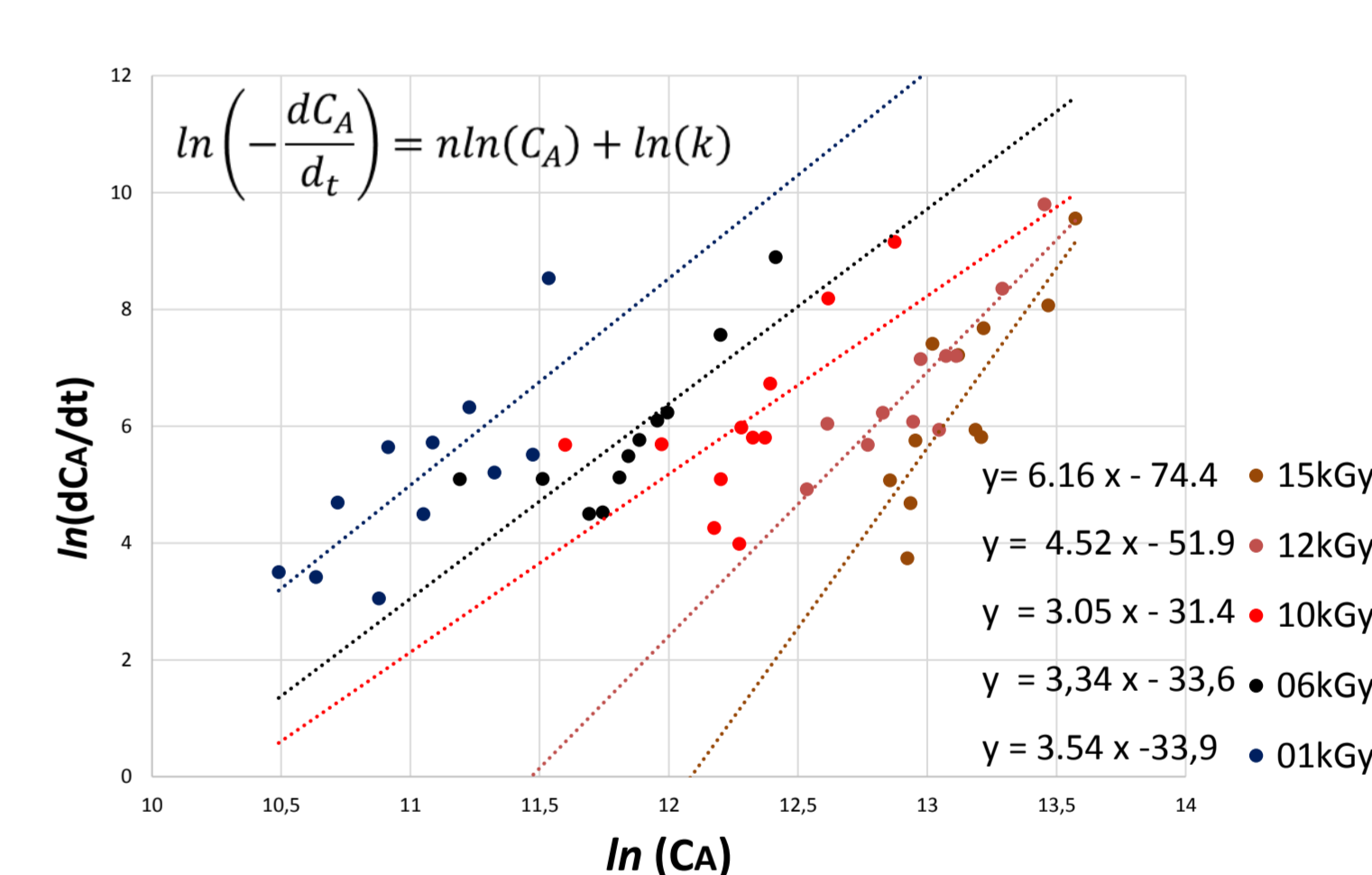
EPR spectrum of cellulose paper - measured at different times after irradiation



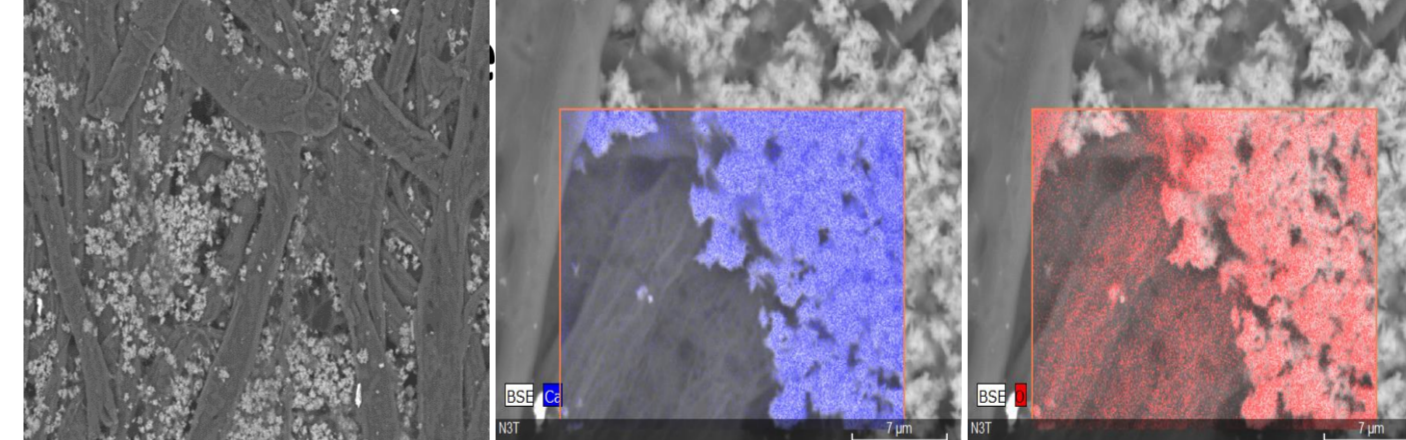
Integrated Spin concentration(CA)



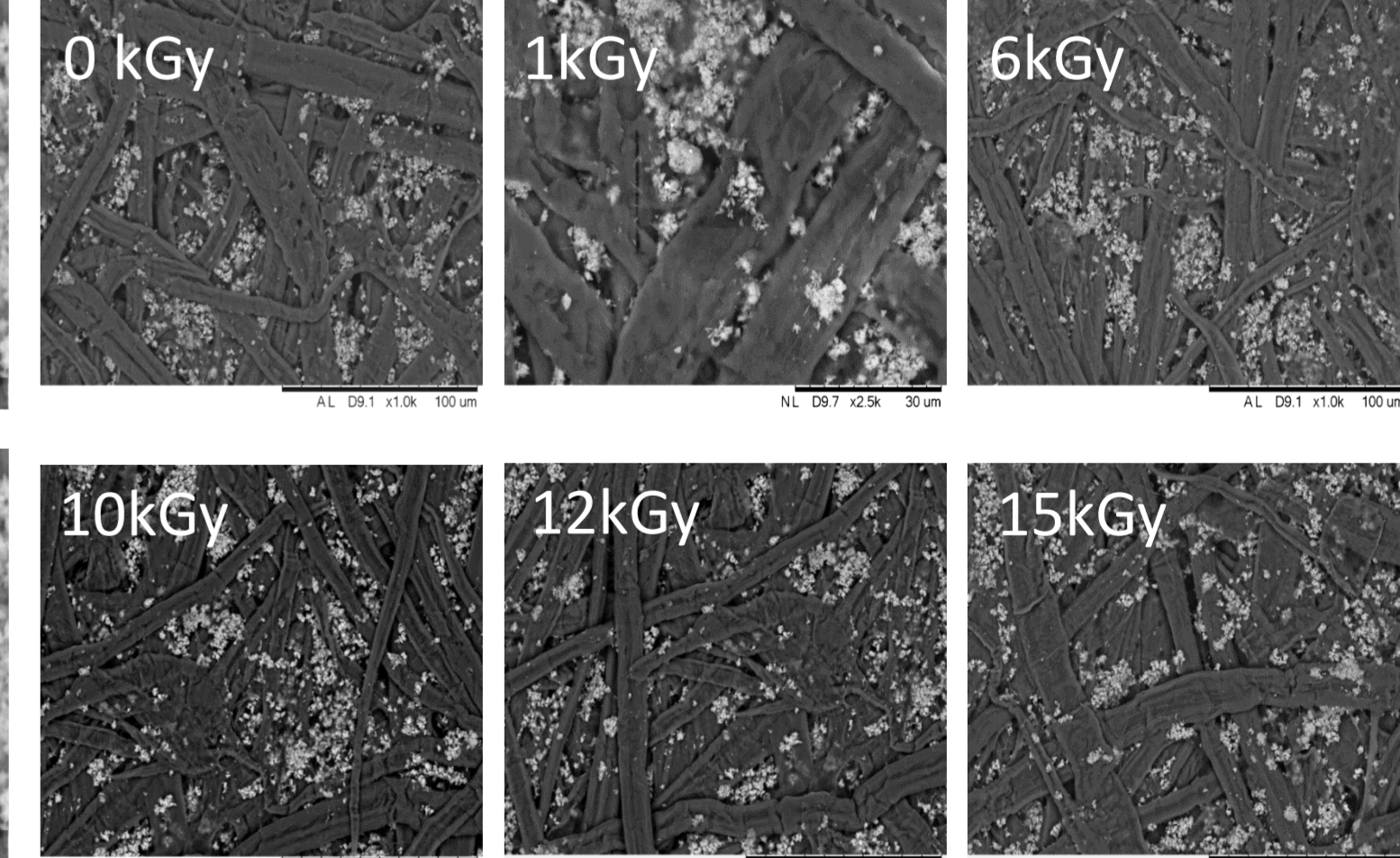
Order of reaction - differential method



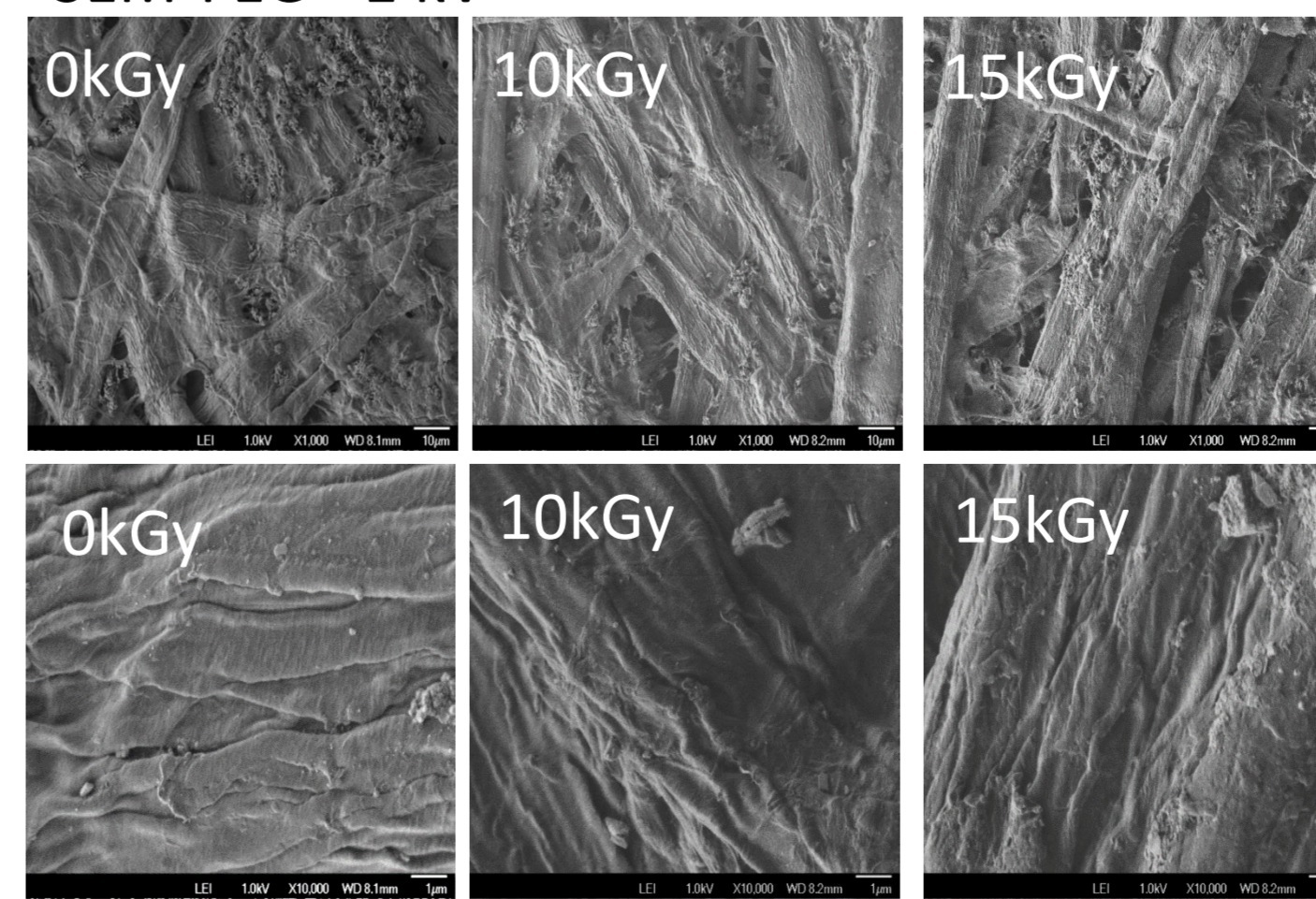
Dispersive Spectrometry(SEM-EDS) Non-irradiated paper- BSE



SEM 15 kV, BSE (backscattered electrons), low vacuum mode



SEM-FEG - 1 kV CaCO₃ (confirmed by XRD)



No morphologic changes detected

ACKNOWLEDGEMENTS

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REFERENCES

- ✓ Kodama, y.; Rodrigues, o. Jr.; Garcia, r. H. L.; Santos, p. S.; Vasquez, P. A. S.. Study of Free Radicals in Gamma Irradiated Cellulose of Cultural Heritage Materials Using Electron Paramagnetic Resonance. *Radiation Physics and Chemistry*, 124, 2016. 169-173
- ✓ Hiromi Kameya and Mitsuko Ukai (2013) Cellulose – Fundamental Aspects, Chapter 12, Analysis of Relaxation Behavior of Free Radicals in Irradiated Cellulose Using Pulse and Continuous-Wave Electron Spin Resonance, <http://dx.doi.org/10.5772/75459>.
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CONCLUSIONS

No significant modifications (side-effects) appear in the irradiated material after the radical decay time, the material will stay stable for the remaining lifetime. Proposed method using electron paramagnetic resonance results showed suitably to study the behavior of radicals on cellulosic based cultural heritage materials