

Original Research

**THERMAL TRANSFORMATION OF NSAID-DERIVED MATERIALS:
MORPHOLOGICAL AND COMPOSITIONAL INSIGHTS**

DOI: 10.5937/EcoTER26427S

**Katarina Stojanović^{1,a*}, Marija Simić^{1,b}, Danka Aćimović^{1,c}, Branislava Savić Rosić^{1,d},
Marija Ječmenica Dučić^{1,e}, Tanja Brdarić^{1,f}**

¹ University of Belgrade, Vinča Institute of Nuclear Sciences-National Institute of the
Republic of Serbia, Mike Petrovića Alasa 12-14, 11000 Belgrade, SERBIA

^a <https://orcid.org/0009-0003-3467-9875> ^b <https://orcid.org/0000-0001-9518-7709>

^c <https://orcid.org/0000-0001-7506-9684> ^d <https://orcid.org/0000-0002-4632-7960>

^e <https://orcid.org/0000-0002-6437-519X> ^f <https://orcid.org/0000-0003-2547-7123>

Abstract

The management and treatment of pharmaceutical waste has recently attracted increasing attention from the scientific and professional community. Residues derived from nonsteroidal anti-inflammatory drugs (NSAIDs) represent carbon-rich precursors suitable for thermal conversion into carbon materials with adaptable structural properties. Moreover, these materials show potential as functional materials due to their high surface area, porosity, and surface functional groups, enabling applications in adsorption and catalysis. The aim of this study was to investigate the influence of calcination temperature on morphological and compositional transformations of NSAID-derived materials. Samples were thermally treated at 400, 600, and 800 °C in air and characterized using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX). SEM analysis revealed pronounced temperature-dependent structural evolution from compact carbonaceous morphology at 400 °C to heterogeneous microstructures containing elongated and needle-like domains at 800 °C. EDX analysis confirmed progressive elemental redistribution with increasing temperature, indicating gradual decomposition of the organic matrix and enrichment of inorganic phases. The obtained results provide insight into the thermal transformation mechanisms of pharmaceutical-derived materials and demonstrate their potential as precursors for functional carbon-based materials.

Keywords: NSAID, calcination, SEM.

INTRODUCTION

The development of sustainable materials derived from waste resources has become an important research direction in modern materials science and environmental engineering, but also a promising source of secondary raw materials. In recent years, considerable attention has been devoted to converting organic waste into functional carbon-based materials through thermal processes such as pyrolysis and calcination, which produce carbon structures with

* katarina.stojanovic@vin.bg.ac.rs

tunable porosity, surface functionality, and morphological features [1,2]. These materials have attracted interest for a wide range of applications, including catalysis, adsorption, energy storage, and electrochemical environmental technologies.

Temperature plays a key role in determining the characteristics of thermally treated materials. Increasing calcination temperature promotes progressive decomposition of the organic matrix, release of volatile components, and formation of new inorganic phases, which can significantly affect the morphology and composition of the resulting materials [3]. Understanding these transformations is essential for evaluating the potential of such materials for further functional applications.

Pharmaceutical compounds represent an interesting, yet still insufficiently explored, precursor for thermally derived materials, as the increasing production of pharmaceuticals has generated significant amounts of pharmaceutical waste that pose a potential environmental risk [4]. Many pharmaceutical molecules contain aromatic rings and heteroatoms that can contribute to the formation of carbon materials with defined morphology during thermal treatment; however, systematic studies addressing the morphological and compositional evolution of pharmaceutical-derived materials during calcination remain limited.

In this study, NSAID-derived material was subjected to calcination at 400, 600, and 800 °C in order to investigate temperature-induced changes in morphology and elemental composition. Morphological characteristics were analyzed using scanning electron microscopy (SEM), while elemental composition was determined by energy-dispersive X-ray spectroscopy (EDX). The aim of this work was to provide insight into the thermal transformation behavior of pharmaceutical-derived materials and to evaluate the influence of calcination temperature on their morphological and compositional evolution.

MATERIALS AND METHODS

The NSAID tablets (2 g; diclofenac Duo, Brufen, and naproxen, Galenika), past their expiration date, were crushed into a powder and thermally treated in a cylindrical furnace in air. The samples were heated at a rate of 10 °C min⁻¹ to 400, 600, or 800 °C, held at the target temperature for 60 min, and then cooled to room temperature under ambient conditions.

A FESEM-EDS system (FEI Scios 2) was used to examine surface morphology and elemental composition. Imaging was performed at an accelerating voltage of 5.0 kV, while EDS spectra were acquired with a collection time of 20 min. High-resolution SEM micrographs and corresponding EDS spectra were obtained from selected micro-areas at a chamber pressure of 2.8×10^{-4} Pa.

RESULTS AND DISCUSSION

SEM analysis revealed pronounced morphological transformations as a function of calcination temperature.

The sample calcined at 400 °C (Figure 1a) exhibits a relatively compact and agglomerated morphology dominated by large irregular particles. A prominent cauliflower-like aggregate can be observed in the central region, surrounded by smaller fragments and plate-like

particles. The surface appears relatively dense with limited visible porosity, suggesting that the organic precursor is only partially decomposed and that the material still retains a compact carbonaceous matrix.

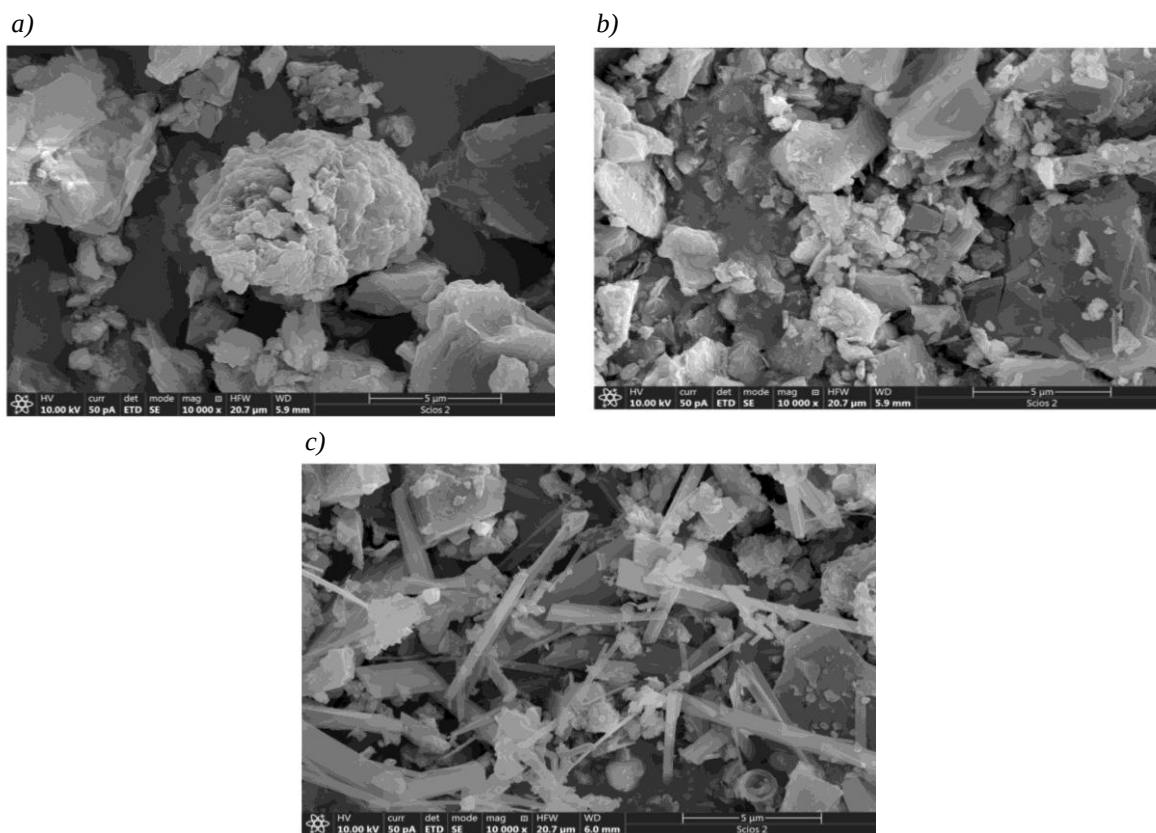


Figure 1 SEM micrographs of NSAID-derived materials calcined at: a) 400 °C; b) 600 °C; and c) 800 °C

A significant structural transformation occurs at 600 °C (Figure 1b). The morphology becomes more fragmented and heterogeneous, consisting of irregularly shaped particles with sharp edges and numerous smaller fragments distributed between larger grains. The structure appears more disordered compared to the 400 °C sample, indicating progressive decomposition of the precursor material and partial disintegration of the original particle aggregates. The increased number of smaller particles suggests release of volatile components and restructuring of the material during thermal treatment.

The material obtained at 800 °C (Figure 1c) shows a markedly different morphology characterized by the formation of elongated rod-like and needle-like structures distributed throughout the sample. These anisotropic microstructures coexist with smaller irregular fragments, resulting in a highly heterogeneous microstructure. The formation of such elongated domains suggests advanced structural reorganization and possible crystallization or growth of inorganic phases at elevated temperature. The presence of these structures significantly increases structural complexity and may contribute to enhanced surface accessibility of the material.

The observed morphological evolution demonstrates that increasing calcination temperature strongly influences the microstructure of NSAID-derived materials. Structural transformation from compact aggregates toward heterogeneous microstructures reflects progressive decomposition of the organic matrix and development of new structural features.

These results confirm that increasing calcination temperature promotes transformation from compact aggregates to heterogeneous microstructures with higher structural complexity.

Table 1 Elemental composition of thermally treated samples (weight %)

Temperature (°C)	C (%)	O (%)	Na (%)	Mg (%)	Si (%)	Cl (%)	Ti (%)
400	43.792	29.776	8.578	5.061	8.797	2.487	1.509
600	11.025	13.915	21.499	14.894	33.527	4.755	0.384
800	35.744	25.951	18.979	1.7250	5.733	10.046	1.823

EDX analysis (Table 1) shows temperature-dependent variations in elemental composition. The 400 °C sample is dominated by carbon and oxygen, consistent with residual organic content. At 600 °C, the relative contribution of inorganic elements increases, indicating further decomposition of the organic matrix. Although the overall composition at 400 °C and 800 °C remains comparable in terms of major elements, the 800 °C sample exhibits higher apparent Na and Cl signals (Table 1). This is attributed to surface redistribution of inorganic species and increased detectability following extensive matrix decomposition, rather than bulk enrichment. At 800 °C, the coexistence of carbon with inorganic elements such as Na, Mg, and Si suggests the presence of multiple inorganic components, consistent with the observed morphological heterogeneity.

CONCLUSION

This study demonstrates that calcination temperature plays a key role in the structural evolution of NSAID-derived materials. Thermal treatment at 400–800 °C induces pronounced morphological and compositional changes. SEM analysis shows a transition from compact carbonaceous structures at lower temperatures to heterogeneous microstructures with elongated and needle-like features at higher temperatures. EDX results further indicate temperature-dependent redistribution of inorganic elements. These combined effects lead to increased structural complexity at elevated temperature, highlighting the importance of thermal treatment in tailoring the properties of pharmaceutical-derived carbon materials. The findings provide insight into their transformation behavior and support their potential use as precursors for functional carbon-based materials.

ACKNOWLEDGEMENT

The authors are grateful to the Ministry of Science, Technological Development and Innovation of the Republic of Serbia for financial support according to the contract with registration No. 451-03-33/2026-03/ 200017.

REFERENCES

- [1] Yuan, X., Dissanayake, P.D., Gao, B., *et al.* J. Environ. Manage. 2021, 296, 113128.
- [2] Ma, Q., Yu, Y., Sindoro, M., *et al.* Adv. Mater. 2017, 29, 1605361.
- [3] Boakye, K., Khorami, M., Ganjian, E., *et al.* Int. J. Emerg. Technol. Innov. 2021, 2, 00016.
- [4] Manole, F., Marian, P., Mekeres, G.M., *et al.* Pharmacophore 2023, 14(2) 106–110.