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PROOF-OF-CONCEPT EXPERIMENT REPORT

ANALYSIS OF SiO_2 AND BaSO_4 LEACHABLES FROM DENTAL
COMPOSITE MATERIAL BY THERMAL LENS SPECTROMETRY

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Proof-of-Concept experiment details

Received sample: Four bottles of liquids containing SiO₂ and BaSO₄ NPs released from artificial teeth under chewing simulation.

Chewing simulation was performed in chewing simulator (moving in Z-axis: 5 mm, frequency 40 mm/s; moving in X-axis: 2 mm, frequency 20 mm/s), total number of cycles: 3 x 120.000 cycles with loads: 3 kg, 5 kg and 9 kg. Chewing simulation is performed with composite teeth in DDH₂O (samples D₁ and L₁) and citric acid solution (samples D₂ and L₂).

Planned analysis:

Application of thermal lens spectrometry (TLS) for determination of the amount of SiO₂ and BaSO₄ NPs released from artificial teeth under chewing simulation.

1st step: UV-vis analysis to provide the optimal condition for TLS measurements

2nd step: Calculation of detection limits (LODs) for SiO₂ and BaSO₄ determination by TLS

3rd step: Determination of SiO₂ and BaSO₄ leachables from artificial teeth (resin based composite material) by TLS

Sample preparation: For the purpose of calculation LODs of the method the calibration curves were constructed by the use of standards solution prepared in DDH₂O with adjusted pH.

Measurement author, dates and place: the sample preparation and the reported measurement has been performed by izr. prof. Dorota Korte at University of Nova Gorica (campus of Rožna Dolina) in the period from July 2021-July 2022.

Preliminary measures: The obtained LOD for SiO₂ and BaSO₄ determination was 30 and 10 ppb, respectively, whereas relative standard deviation (RSD) was between 1-5% what indicates high sensitivity and good reproducibility of the method.

It was found that the release of SiO₂ from the designed resin-based composites is at the level of LOD of the detection method, whereas BaSO₄ under LOD. It can be also stated that the designed resin-based composites containing SiO₂ MPs and NPs is a promising dental material with improved mechanical properties such as enhanced hardness and scratch resistance since any leaching neither of SiO₂ and BaSO₄ NPs from artificial teeth under long term chewing simulation was observed. Thus, such material seems to be an excellent choice as dental resins especially in case of large area restoration where the mechanical stresses under use is of large value.

Observation conditions:

TLS: excitation beam wavelength 405 nm; excitation beam power 200 mW. Probe beam wavelength 633 nm, modulation frequency 8 Hz

Main aim of the proposal:

Nowadays ceramics and resin-based dental composites containing micro (MPs) and nanoparticles (NPs) are used for restoring dental caries or other defects instead of conventional alloys due to the improved mechanical properties (strength, toughness, surface hardness, durability) of the whole matrix. This is of high importance especially in case of large area restorations. Furthermore, the resin-

based dental composites are the most popular restorative materials and are mainly composed of resin polymeric matrix, inorganic fillers particles and silane coupling agents. In the present study, inorganic filler in the resin-based composite is micro-sized SiO₂ with traces of nano-sized SiO₂. Additionally, the resin-based material contains small amounts (few tenths of percent) of nano-sized BaSO₄ used as a functional additive during production. Incorporation of these two components in dental materials introduces the oral route of exposure for these nanomaterials (NMs) [Y. Liu, Y. Sun, F. Zeng, X. Weili, Y. Liu, L. Geng, Effect of nano SiO₂ particles on the morphology and mechanical properties of POSS nanocomposite dental resins, *J. Nanopart. Res.* 16 (2014) 2736-2744. <https://doi.org/10.1007/s11051-014-2736-0>], [L.H. Prentice, M.J. Tyas, M.F. Burrow, The effect of ytterbium fluoride and barium sulphate nanoparticles on the reactivity and strength of a glass-ionomer cement. *Dent. Mater.* 232(8) (2006) 746-751. <https://doi.org/10.1016/j.dental.2005.11.001>]. Their absorption in the gastrointestinal tract may be high due to their small particle size causing histopathological changes in liver and kidneys or alterations in blood parameters. Furthermore, oral uptake of SiO₂ and BaSO₄ NPs from dental materials is likely to occur at low doses over long periods of time leading to their accumulation in body tissues [A.T. Florence, Nanoparticle uptake by the oral route: fulfilling its potential? *Drug Discov. Today: Technol.* 2 (2005) 75–81. <https://doi.org/10.1016/j.ddtec.2005.05.019>]. Thus, monitoring of their leachables requires highly sensitive detection techniques. Because of that the goal of the analysis was the monitoring of the leachables from composite resin-based dental material containing micro (MPs) and nanoparticles (NPs) that are used for replacing the missing teeth as part of denture by the use of TLS.

Results

UV-vis measurements

Both SiO₂ and BaSO₄ samples show high absorption in the UV range of the spectra whereas a small resonance peak around 400 nm resulting from the presence of NPs.

TLS analysis:

The SiO₂ and BaSO₄ determination was performed by the use of calibration curve presented in Fig. 1

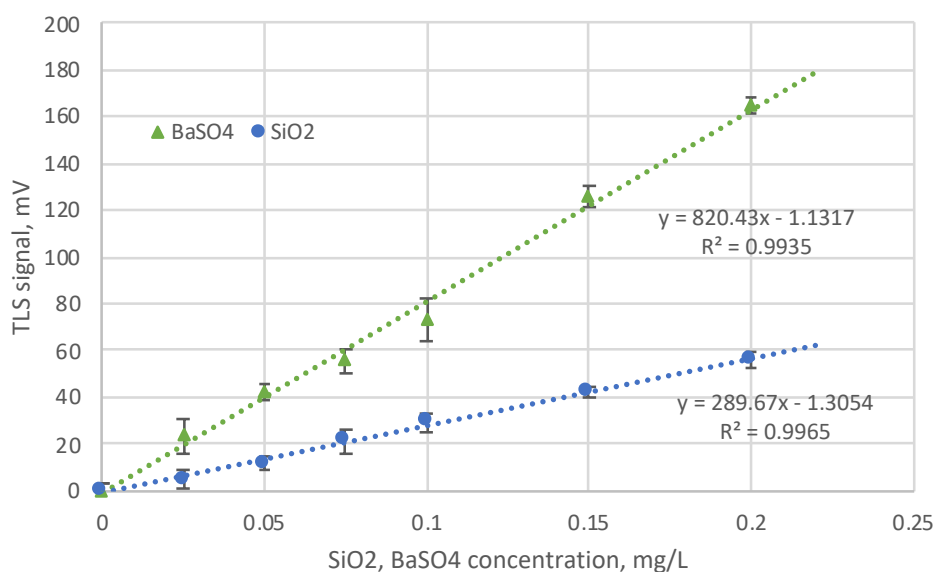


Fig. 1. Calibration curves used in the study for SiO₂ and BaSO₄ determination.

The obtained LOD for SiO₂ and BaSO₄ determination was 30 and 10 ppb, respectively, whereas RSD was between 1-5% what indicates high sensitivity and good reproducibility of the method.

Table 1. Values of SiO₂ and BaSO₄ leachables from composite resin-based dental material obtained in the study.

Sample no.	SiO ₂ /ppb	BaSO ₄ /ppb
D1	31	ND*
L1	28	ND
D2	36	9
L2	33	7

*not detected

It was found that the leaching of SiO₂ from the designed resin-based composites is at the level of LOD of the detection method, whereas BaSO₄ under LOD.

Summary

- the designed resin-based composites containing SiO₂ and BaSO₄ MPs and NPs are promising dental materials with improved mechanical properties such as enhanced hardness and scratch resistance since, at least according to these measurement results, they do not release neither SiO₂ and BaSO₄ NPs from artificial teeth under long term chewing simulation.
- such material seems to be an excellent choice as dental resins especially in case of large area restoration where the mechanical stresses under use is of large value.

Summary about the analytical capability of TLS in these experimental conditions:

- TLS is an effective tool in the analysis of leachables from composite resin-based dental materials containing micro (MPs) and nanoparticles (NPs).
- The obtained LOD for SiO₂ and BaSO₄ determination was 30 and 10 ppb, respectively what indicates high sensitivity of the method.
- lower LODs are possible to achieve by the use of excitation wavelength of wavelength in the UV region of the spectra
- The obtained RSD was between 1-5% what indicates good reproducibility of the method.

This report has been written by Dorota Korte (Nova Gorica, 15 July 2022)