

Abstract

Heat is one of the fundamental factors determining the nature of substances. It affects the internal structure and composition of every material, changing its physical and chemical properties. Various thermal analyses contribute to a deeper understanding of the behavior of matter at different temperatures. One such analysis is thermo-Raman spectroscopy, a method that tracks phase changes in solids, liquids and gases as a function of temperature. However, this method usually requires special equipment in the form of a benchtop spectrometer equipped with a high-temperature heating cell. The aim of this thesis was to develop, construct and test an experimental apparatus enabling *in-situ* monitoring of solid-state phase transitions using relatively accessible portable Raman spectrometers. The apparatus consists of a heating device and a portable spectrometer (532 nm) with modified optics, allowing measurements from a safe distance. To verify the functionality of the apparatus, six experiments were conducted. Silicon carbide, potassium nitrate and four hydrated sulfates were chosen as reference materials. The results confirmed that the spectrometer can track phase changes very well and is not affected by thermal radiation. The design of the heating device allows heat transfer only through the sample holder, which may lead to uneven heat distribution in the sample associated with phase heterogeneity. As a result, a longer time is needed to temper the samples and complete the phase transitions. For all experiments with hydrated sulfates, this was confirmed by subsequent powder X-ray diffraction analysis, which determined the presence of both dehydrated and hydrated phases. The main limitation of using the apparatus for standard thermal analysis is the access of atmospheric oxygen to the sample. This significantly narrows the range of usable samples that do not undergo oxidation even at elevated temperatures. When using non-reactive samples, this alternative methodology can provide valuable information on the phases involved in the thermal process. This methodology can find extensive use in simulating thermally active oxidizing environments, such as certain types of fumaroles or the surface of burning-coal waste piles.