A novel test method for measurement of soluble alkalis from supplementary cementitious materials (SCMs) is proposed. For this purpose, the concentration of SCMs’ soluble sodium in 1 N Potassium Hydroxide solution was investigated over a period of 90 days. Similarly, the soluble Potassium was evaluated in a 1 N Sodium Hydroxide, as the host solution. ICP-AES spectral analysis was implemented to determine the targeted ion’s concentration. The results of this new test method were compared with X-ray fluorescence analysis of SCMs to reveal the portion of SCMs’ alkali content that is soluble in a high-pH solution. Eleven non-traditional and natural pozzolans were investigated, in four groups including calcined clays, natural pozzolans, ground bottom ashes, and fluidized bed combustion ashes. Available alkali content of SCMs was also evaluated as per ASTM C311 standard test method.

Moreover, for further assessment of SCMs’ contribution to the alkalinity of concrete pore solution, cement pastes incorporating 20% SCM were prepared, and the pore solution was extracted from the fresh paste, as well as at 7 and 28 days of curing. The contribution of SCM incorporation to the cement pore solution alkalinity was compared with ASTM C311 standard test method. The results of the soluble alkali test method revealed that 15 to 85% of SCMs’ total alkali content is soluble at 90 days. The alkali dissolution graph over time plateaued after 28 to 90 days, showing the age at which the maximum soluble alkali content of SCMs is reached. ASTM C311 test results showed volcanic ashes have the highest available alkalis content compared to other investigated SCMs. Also, no linear relationships between the total and available alkali content of SMCs were observed. Calcined clays had the lowest available alkali content based on the results of cement paste pore solution extraction analysis.