The presented thesis provides an experimental study of the K-shell double vacancy production in the electron capture decays of ^{55}Fe, ^{54}Mn, ^{65}Zn using a pair of Timepix3 detectors. Measured data are preprocessed and explored. The methodology for the calculation of the probability of K-shell double vacancy production in ^{55}Fe and ^{54}Mn is developed. Therefore, an extensive amount of signal and background processes were considered during the development of the methodology. The measurement setup is defined and optimized in the Allpix^2 framework for a simulation of detection efficiencies of particles participating in the signal and background processes. The probability of K-shell double vacancy creation in the electron capture decay of ^{55}Fe was measured to be $P_{\mathrm{KK}} = (1.406 \pm 0.05) \times 10^{-4}$ with a systematic error of $\Delta_{\mathrm{sys}}(P_{\mathrm{KK}}) = ^{+0.030}_{-0.034} \times 10^{-4}$. The value of P_{KK} for the electron capture decay of ^{54}Mn found to be $P_{\mathrm{KK}} = (3.93 \pm 0.44) \times 10^{-4}$ with a systematic error of $\Delta_{\mathrm{sys}}(P_{\mathrm{KK}}) = ^{+0.25}_{-1.11} \times 10^{-4}$. Resulting probabilities for K-shell double vacancy production in ^{55}Fe and ^{54}Mn are in agreement with the latest results.