In this study, the effects of the combination of acid hydrolysis and Donnan dialysis on the high-quality struvite production from digested sewage sludge were examined. The Box–Behnken design was applied in the hydrolysis using oxalic acid for the optimization of the conditions that affect the nutrients and metal release from digested sludge. An optimal condition was obtained at oxalic acid 0.5 M, acid/sludge ratio (mL/g) 10/1, and reaction time 60 min. The separation of the metals from hydrolyzed sludge liquid was carried out with the Donnan dialysis using a Nafion 117 cation exchange membrane. At the end of the 4 hours of operating time, the recovery values for Zn, K, Na, Mg, Fe, and Al were obtained as 67.9%, 62.1%, 57.6%, 39.4%, 5.3%, and 2.5%, respectively.