

## Article

# Systematic Approach to the Synthesis of Cobalt-Containing Polyoxometalates for Their Application as Energy Storage Materials

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**Abstract:** New energy storage materials are an object of study within the framework of the global energy transition. The development of renewable sources is being boosted thanks to stationary energy storage systems such as redox flow batteries (RFBs). This work reports the synthesis of the cobalt-containing Keggin-type polyoxometalates  $[\text{CoW}_{12}\text{O}_{42}]^{6-}$  ( $\text{CoW}_{12}$ ) and  $[\text{Co}(\text{H}_2\text{O})(\text{SiW}_{11}\text{O}_{39})]^{8-}$  ( $\text{CoSiW}_{11}$ ), which have previously been shown to have applicability in RFBs. These procedures were reassessed to meet the strict requirements associated with the further implementation of RFBs, including fast and affordable synthetic procedures with high reaction yields. In contrast to the lengthy and complicated synthetic approaches published to date, the optimized synthesis reported in this work enables the isolation of the pure crystalline salt of the  $\text{CoW}_{12}$  anion with a 75% reduction of the time of the whole reaction procedure, eliminating tedious steps such as the recrystallization and including a 20% increased yield. The control of the stoichiometry, fine-tuning of reaction conditions, and the identification of intermediate species, as well as the acidic equilibria taking place during the process, were monitored via thermal, spectroscopic, and structural analyses. In the case of the  $\text{CoSiW}_{11}$  anion, its preparation was based on a simple and highly efficient procedure. Moreover, promising electrochemical properties were observed with the use of the one-pot synthetic approach, in which the stoichiometric amounts of the starting reagents are dissolved in the supporting electrolyte to be directly implemented as the electrolyte for a RFB.

**Keywords:** polyoxometalate; cobalt; synthesis; energy storage materials; electroactive species; electrolyte



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## 1. Introduction

Polyoxometalates (POMs) are a rich family of inorganic metal–oxygen clusters that constitute a unique class of compounds [1]. Due to their special reactivity together with their structural and compositional versatility, they are employed in different areas such as materials science [2], catalysis [3], and medicine [4]. The simplest procedures for the synthesis of POMs start from aqueous solutions of  $[\text{MO}_4]^{n-}$  ( $\text{M} = \text{V}, \text{Mo}, \text{W}$ ) oxyanions that undergo complex self-assembly processes via acidic condensation equilibria of  $[\text{MO}_4]$  polyhedral subunits until the formation of the desired polyanion is achieved [5]. Afterwards, clusters are precipitated by the addition of a suitable counterion (e.g., alkali metals, organic cations). These complex formation equilibria are highly influenced by chemical and physical stimuli; hence, their fine-tuning is a key factor in the control of the formation of the desired POMs with high yield and purity [6]. Extensive research has been carried out over the years on the optimization of the synthetic procedures for the formation of POMs [7]. Consequently, long lists of variables that are crucial for the formation of given clusters have been identified. These include concentration and type of the metal source, ionic strength and pH, sequence of the addition of reagents, and external physical (e.g.,

# An Approach For Systematic Evaluation Of Materials For Structural Application

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