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Preparation and investigation of freeze dried γ-cyclodextrin metal-organic frameworks

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Cyclodextrin-based metal-organic frameworks (also known as CD-MOFs) are highly porous materials with non-toxic characteristics that are formed by coordinate bonding between alkali metal cations and cyclodextrins. In addition to absorbing poorly water-soluble drugs, CD-MOFs can also modify the physicochemical characteristics of these molecules. Research on CD-MOFs is still in its infancy, but their potential for use in delivery-particularly in the lung-seems promising. The traditional process of creating CD-MOF crystals by vapor diffusion (VD) is time-consuming and unsuitable for mass production. Our aim was to use the freeze drying (FD) method in one step to create a novel approach for the preparation of CD-MOFs. MOFs consisting of γ-cyclodextrin (γCD) and potassium cations were employed to encapsulate the poorly water-soluble model drug Ibuprofen (IBU). Using the LeanQbD® software, we designed the experiments based on the Quality by Design (QbD) concept. Light- and Scanning Electron Microscope (SEM) was utilized to observe the morphology and particle size of the FD and control VD crystals. The size of the particles was measured by Laser Diffraction. In addition, the products were characterized by Differential Scanning Calorimetry (DSC), X-ray Powder Diffraction (XRPD) and Fourier Transform Infrared Spectroscopy (FT-IR). According to QbD, we identified the 3 most critical factors, which were the molar ratio of the IBU to the γCD, reaction time, and the percentage of the organic solvent. Depending on various parameters, the SEM pictures showed variously shaped and surfaced particles. Among the prepared samples, two samples seemed promising, differing only in their reaction times. They had a spherical shape, and their d(0.5) values were 3.2 µm and 3.9 µm. Based on DSC and FT-IR results, we concluded that a γCD-MOF/IBU complex was also formed using the freeze drying method. The complex showed new crystal structures, as the XRPD results demonstrate. Our formulations could be suggested for further investigations, while γCD-MOFs were expected to be promising carriers for IBU delivery by pulmonary route.

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