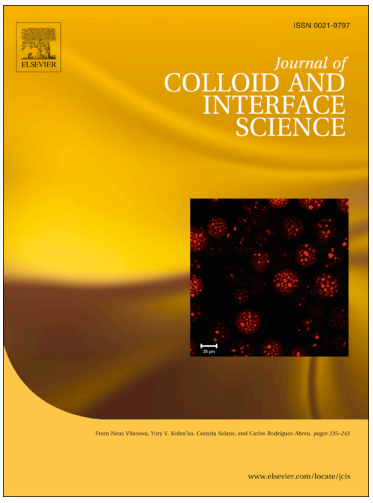

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Abstract: Significant research has been focused on the synthesis of metal-organic frameworks (MOFs) with controllable compositions and structures, while much fewer works have been devoted to the construction of large micro-sized MOFs with uniform sizes and morphologies, which could be beneficial for practical applications. In this paper, a unique microfluidic jet spray drying technology has been adopted to reassemble nano-sized MIL-101 building blocks into hierarchical microparticles with uniform and large particle sizes. Specifically, suspension precursors of nano-sized MIL-101 building blocks are atomized into uniform droplets and then converted to microparticles on a one-to-one basis through a fast and scalable spray drying process. The particle size and morphology can be controlled by adjusting the solid concentration of the suspension and the drying temperature. The particle formation process with evolution of different morphologies are discussed. The resultant uniform MIL-101 microparticles possess hierarchical porosities and maintain the intrinsic crystal structure, microporosity and thermal stability of the nano-sized building blocks. They demonstrate a high efficiency toward benzene adsorption from n-octane solutions with high adsorption rates and very high adsorption capacities under batch conditions. Moreover, the large particle size and hierarchical structure make them applicable as filler of a fixed bed for dynamic flow separation of benzene from n-octane solutions with promising performance. The microfluidic jet spray drying technology can also be extended for the reassembly of other uniform MOF microparticles.