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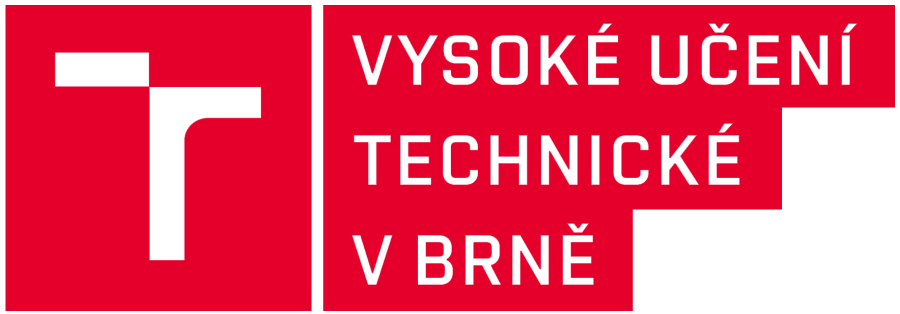

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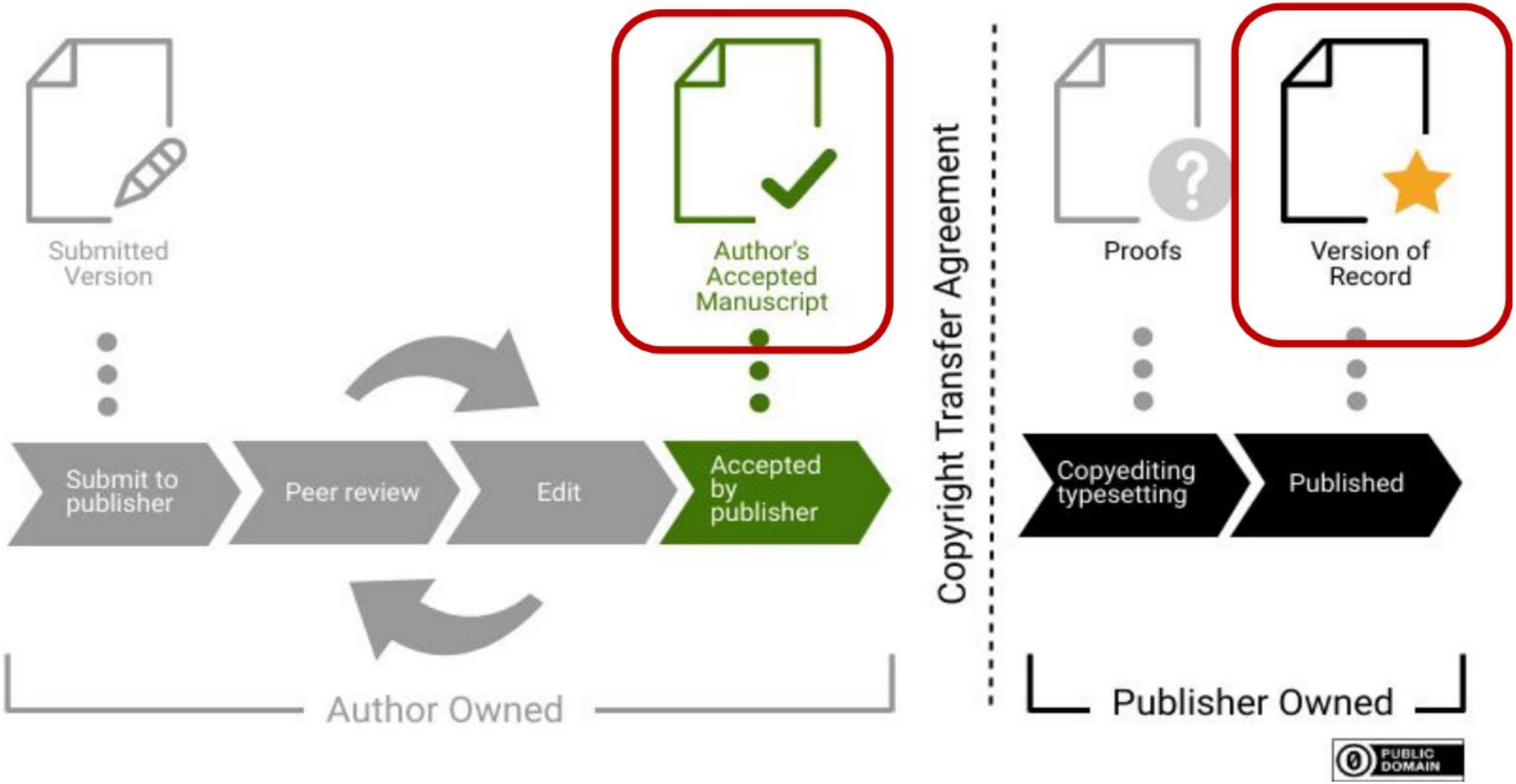
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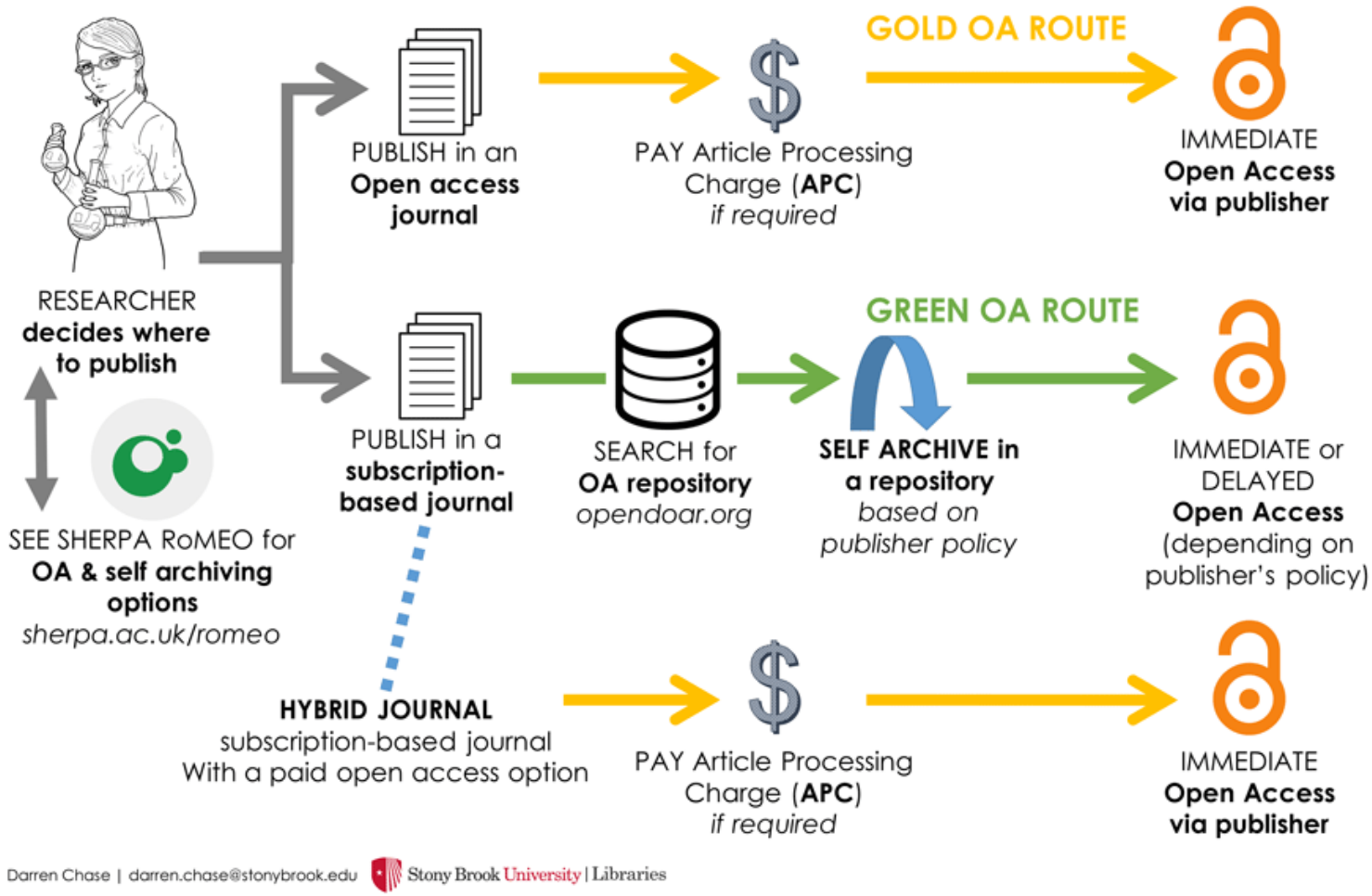

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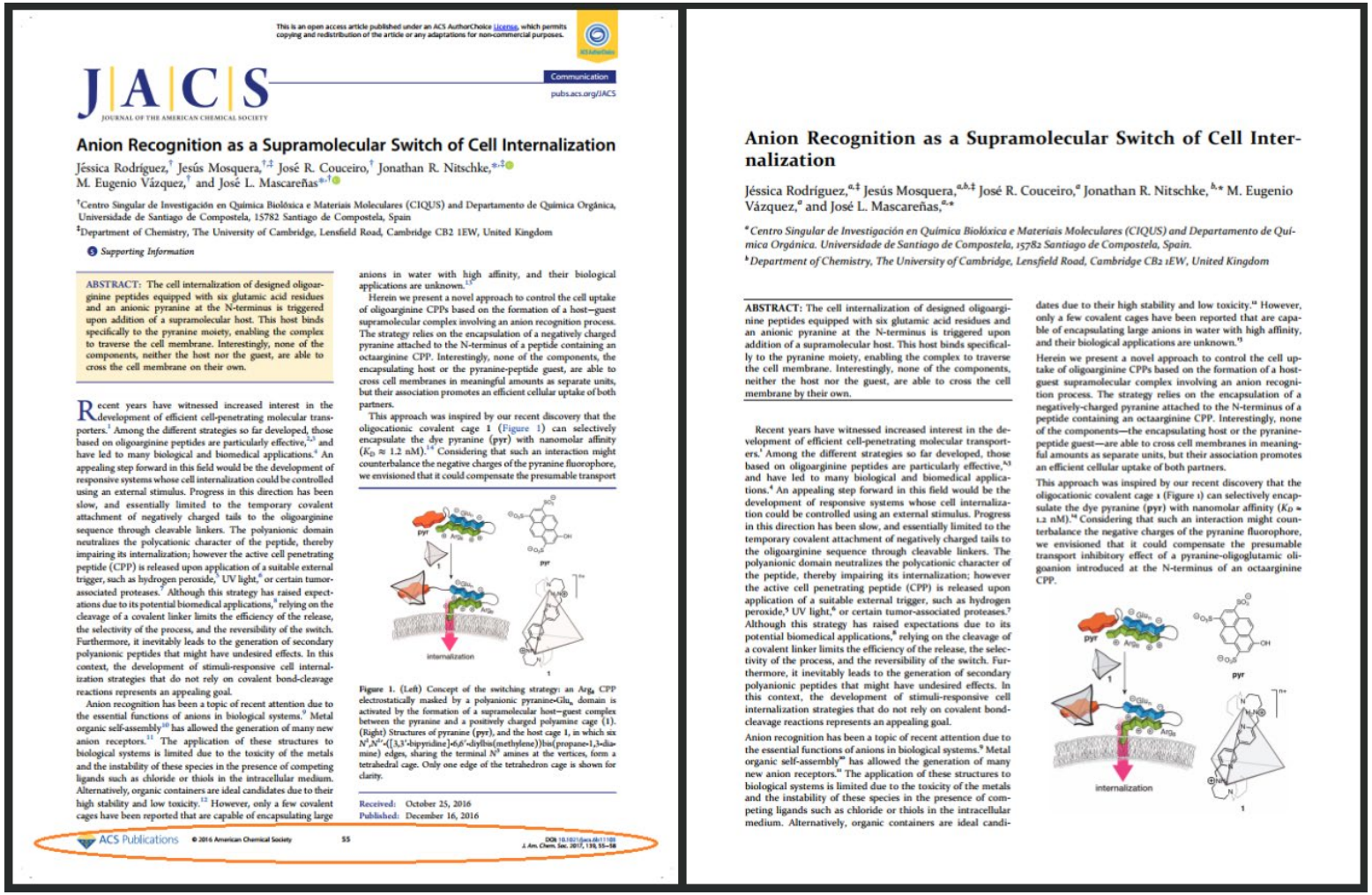

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Anion Recognition as a Supramolecular Switch of Cell Internalization

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Supporting Information

ABSTRACT: The cell internalization of designed oligoarginine peptides equipped with six glutamic acid residues and an anionic pyranine at the N-terminus is triggered upon addition of a supramolecular host. This host binds specifically to the pyranine moiety, enabling the complex to traverse the cell membrane. Interestingly, none of the components, neither the host nor the guest, are able to cross the cell membrane on their own.

Recent years have witnessed increased interest in the development of efficient cell-penetrating molecular transporters.[1] Among the different strategies so far developed, those based on oligoarginine peptides are particularly effective,[2,3] and have led to many biological and biomedical applications.[4] An appealing step forward in this field would be the development of responsive systems whose cell internalization could be controlled using an external stimulus. Progress in this direction has been slow, and essentially limited to the temporary covalent attachment of negatively charged tails to the oligoarginine sequence through cleavable linkers. The polyanionic domain neutralizes the polycationic character of the peptide, thereby impairing its internalization; however the active cell penetrating peptide (CPP) is released upon application of a suitable external trigger, such as hydrogen peroxide,[5] UV light,[6] or certain tumor-associated proteases.[7] Although this strategy has raised expectations due to its potential biomedical applications,[8] relying on the cleavage of a covalent linker limits the efficiency of the release, the selectivity of the process, and the reversibility of the switch. Furthermore, it inevitably leads to the generation of secondary polyanionic peptides that might have undesired effects. In this context, the development of stimuli-responsive cell internalization strategies that do not rely on covalent bond-cleavage reactions represents an appealing goal.

Anion recognition has been a topic of recent attention due to the essential functions of anions in biological systems.[9] Metal organic self-assembly[10] has allowed the generation of many new anion receptors.[11] The application of these structures to biological systems is limited due to the toxicity of the metals and the instability of these species in the presence of competing ligands such as chloride or thiols in the intracellular medium. Alternatively, organic containers are ideal candidates due to their high stability and low toxicity.[12] However, only a few covalent cages have been reported that are capable of encapsulating large anions in water with high affinity, and their biological applications are unknown.[13]

Herein we present a novel approach to control the cell uptake of oligoarginine CPPs based on the formation of a host−guest supramolecular complex involving an anion recognition process. The strategy relies on the encapsulation of a negatively charged pyranine attached to the N-terminus of a peptide containing an octaarginine CPP. Interestingly, none of the components, the encapsulating host or the pyranine-peptide guest, are able to cross cell membranes in meaningful amounts as separate units, but their association promotes an efficient cellular uptake of both partners.

This approach was inspired by our recent discovery that the oligocationic covalent cage **1** (Figure 1) can selectively encapsulate the dye pyranine (**pyr**) with nanomolar affinity ($K_D \approx 1.2$ nM).[14] Considering that such an interaction might counterbalance the negative charges of the pyranine fluorophore, we envisioned that it could compensate the presumable transport

Figure 1. (Left) Concept of the switching strategy: an Arg_8 CPP electrostatically masked by a polyanionic pyranine-Glu_4 domain is activated by the formation of a supramolecular host−guest complex between the pyranine and a positively charged polyamine cage (**1**). (Right) Structures of pyranine (**pyr**), and the host cage **1**, in which six $N^3,N^{3'}$-([3,3'-bipyridine]-6,6'-diylbis(methylene))bis(propane-1,3-diamine) edges, sharing the terminal N^3 amines at the vertices, form a tetrahedral cage. Only one edge of the tetrahedron cage is shown for clarity.

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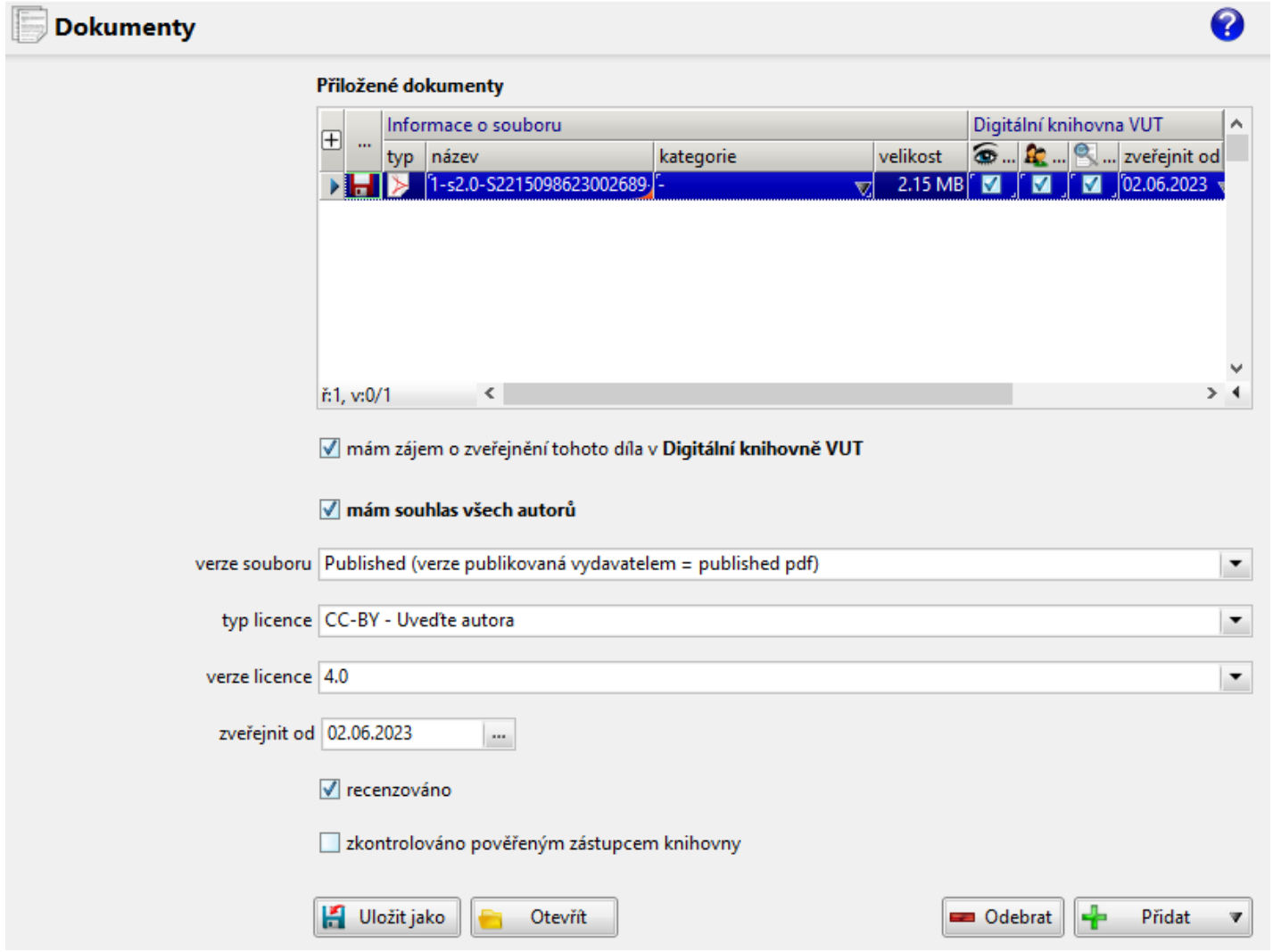

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Abstrakt

Microplastics, which comprise one of the omnipresent threats to human health, are diverse in shape and composition. Their negative impacts on human and ecosystem health provide ample incentive to design and execute strategies to trap and degrade diversely structured microplastics, especially from water. This work demonstrates the fabrication of single-component TiO2 superstructured microrobots to photo-trap and photo-fragment microplastics. In a single reaction, rod-like microrobots diverse in shape and with multiple trapping sites, are fabricated to exploit the asymmetry of the microrobotic system advantageous for propulsion. The microrobots work synergistically to photo-catalytically trap and fragment microplastics in water in a coordinated fashion. Hence, a microrobotic model of "unity in diversity" is demonstrated here for the phototrapping and photofragmentation of microplastics. During light irradiation and subsequent photocatalysis, the surface morphology of microrobots transformed into porous flower-like networks that trap microplastics for subsequent degradation. This reconfigurable microrobotic technology represents a significant step forward in the efforts to degrade microplastics.

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