# Recent Developments in Artificial Molecular-Machine-Based Active Nanomaterials and Nanosystems

Tony Jun Huang

#### Abstract

Artificial molecular machines capable of converting chemical, electrochemical, and photochemical energy into mechanical motion represent a high-impact, fast-growing field of interdisciplinary research. These molecular-scale systems utilize a "bottom-up" technology centered upon the design and manipulation of molecular assemblies and are potentially capable of delivering efficient actuation at length scales dramatically smaller than traditional microscale actuators. As actuation materials, molecular machines have many advantages, such as high strain (40%–60%), high force and energy densities, and the capability to maintain their actuation properties from the level of a single molecule to the macroscale. These advantages have inspired researchers to develop molecular-machine–based active nanomaterials and nanosystems, including electroactive and photoactive polymers. This article will discuss the structures and properties of artificial molecular machines, as well as review recent progress on efforts to move molecular machines from solution to surfaces to devices.

#### Introduction

Artificial molecular motors and machines have recently generated tremendous interest in the scientific community.<sup>1-5</sup> The idea to create artificial molecular machines was originated by Richard Feynman, one of the great physicists and science educators of the 20th century. In his historic address to the American Physical Society, "There's Plenty of Room at the Bottom,"<sup>6</sup> he contemplated,

"What are the possibilities of constructing molecular-scale mechanical machines... What would be the utility of such machines? Who knows? I cannot see exactly what would happen, but I can hardly doubt that when we have some control of the arrangement of things on a molecular scale, we will get an enormously greater range of possible properties that substances can have, and of the different things we can do." $^{6}$ 

In the 1980s, the earliest examples of artificial molecular-level machines were reported, and most were based on the photoisomerization of azobenzene.<sup>7</sup> Since then, the development of synthetic molecular machines has become one of the most intriguing topics in the field of nanoscience and nanotechnology.

Employing the concepts of self-assembly and molecular recognition, Stoddart and other researchers pioneered the creation of a family of mechanically interlocked molecules known as bistable rotaxanes.<sup>1,3,4</sup> These molecules are among the most successful artificial molecular machines constructed to date. The structural formula and graphical representation of a bistable rotaxane R1 are shown in Figure 1. R1 consists of a tetracationic cyclobis (paraquatp-phenylene) (CBPQT4+) ring component as well as a linear rod section. This section contains two different components, a tetrathiafulvalene (TTF) unit and a dioxynaphthalene (DNP) unit. When R1 is at its initial state, the molecule has its TTF unit encircled by the positively charged ring. Upon oxidation, the TTF unit becomes dicationic (TTF2+) and experiences electrostatic repulsion from the ring, causing the ring to shuttle toward the DNP unit in the oxidized state. Conversely, reduction of the TTF<sup>2+</sup> to a neutral TTF unit causes the **R1** to return to its starting state.

The oxidation and reduction of bistable rotaxanes can be realized by applying an electric field, light, or a chemical stimulus.<sup>8–11</sup> Thus, bistable rotaxane, with its two reversible states involving ring translation upon stimulation, can serve as an ideal molecular component of electroactive/photoactive polymers.

There are multiple incentives to developing molecular-level electroactive/ photoactive polymers. For example, as actuation materials, artificial molecular machines such as bistable rotaxanes have at least six advantages:

1. Molecular-machine–based electroactive/ photoactive polymers can maintain their actuation properties across multiple scales (from the single-molecule level to the macroscale), while it is difficult for many other electroactive/photoactive polymers to possess these properties at the microand nanoscales. Rather, these polymers have bulk properties that require multiunit interactions, and thus they are not desirable for molecular-level devices.<sup>12–14</sup> 2. They can generate strains as high as 40%–60%.<sup>1,2</sup>

3. They are characterized by high force and energy densities. For comparison, a bistable rotaxane can generate roughly 100 pN of force,<sup>15</sup> while a kinesin bio-motor, which is much larger than a bistable rotaxane, can generate only 6 pN.<sup>16,17</sup>

4. These molecules can be made metastable (i.e., the molecules exist permanently in their switched state unless another stimulus is applied), a characteristic that saves energy.<sup>18</sup>

5. They can be customized and optimized, thereby conferring the flexibility necessary for a multitude of engineering applications. For example, they can be functionalized with disulfide tethers to facilitate the formation of self-assembled monolayers (SAMs),<sup>19</sup> prepared with amphiphilic (having both hydrophilic and hydrophobic) properties to facilitate the formation of



Figure 1. Molecular structure and schematic representations of a redox-controllable bistable [2]rotaxane **R1**. DNP is dioxynaphthalene; TTF is tetrathiafulvalene; and CBPQT<sup>4+</sup> is cyclobis(paraquat-*p*-phenylene).

Langmuir–Blodgett (LB) films,<sup>20,21</sup> or functionalized within a polymer matrix.<sup>22,23</sup> 6. They can exist over a range of temperatures (from –30°C to 100°C) and pH values (from pH 5 to pH 9), while biomotors are restricted to physiological conditions ( $T \sim$ 37°C, pH ~ 7).<sup>24–26</sup> This last characteristic demonstrates that although biomotors are excellent actuators in human bodies, artificial molecular machines such as bistable rotaxanes are more suitable "molecular actuators" for synthetic devices.

Despite the potential of artificial molecular machines, most of the research carried out so far has focused on molecular behavior in solution.<sup>1,2</sup> However, a solution contains a large number of randomly distributed molecules that cannot be addressed individually or expressed coherently, traits that are required for devices. Therefore, to enable the use of artificial molecular-machine-based electroactive/photoactive polymers in device applications, it is imperative to have these nanoscale machines self-organize at interfaces so that they can be addressed at the nanoscale level. This organization will coordinate their performance on larger scales (microscopic, mesoscopic, and macroscopic). In view of the significance of artificial molecular machines and their behavior on surfaces and in device settings, this article reviews recent efforts to move molecular machines from solution to surfaces to devices.

#### Operating Molecular Machines on Surfaces

In order to incorporate molecular machines into devices, it is imperative that the mechanical switching properties of molecular machines be preserved on surfaces or other solid-state interfaces, where the mechanical movements of the molecules can be harnessed in a cooperative fashion and transmitted to solid-state devices. This section summarizes the recent advances in switching bistable rotaxanes within various solid-state physical environments (e.g., LB films, SAMs, and polymer matrices). Since most applications rely on solid media to transmit actuation forces, these results pave the way for the future development of artificial molecular-machine–based electroactive/photoactive polymers and other functional materials and devices.

#### Closely Packed Langmuir–Blodgett Films

The Langmuir-Blodgett technique<sup>20,27</sup> constitutes an efficient means of transferring organized monolayers from the air-water interface onto a solid substrate. Using this manufacturing approach, one can selforganize and orient amphiphilic bistable rotaxanes.<sup>28,29</sup> In addition, these particular motor molecules in LB films can be considered to be in a liquid-crystalline-like environment and therefore should maintain their mechanical switching properties. The combination of controlling the orientation of the superstructure in the solid state with the switching behavior observed in the solution state makes the LB technique ideal for establishing the interface between amphiphilic bistable rotaxanes and solid supports. Using x-ray photoelectron spectroscopy (XPS), Huang and colleagues demonstrated that amphiphilic bistable rotaxanes R2 are mechanically switchable in closely packed LB films on solid substrates.<sup>30</sup> In XPS, the photoemission intensity of each element depends on the depth at which the photoelectron is emitted; this intensity attenuates exponentially with depth. Molecular switching behavior was monitored by using XPS to track nitrogen, which is present solely in rotaxane's moving part, the ring component. Figure 2 demonstrates that the



Figure 2. Chemical switching of an amphiphilic bistable [2]rotaxane **R2** in a Langmuir–Blodgett film, as monitored by x-ray photoelectron spectroscopy of the nitrogen 1*s* orbital.

nitrogen intensity (shown in red) was significantly higher in the switched state (when the molecules are oxidized) than in the starting state (shown in green). Results of other independent studies involving control experiments and quantitative analysis have confirmed that these observations are consistent with the ring components switching completely from their lower recognition sites (starting state) to the upper ones (switched state).

#### Self-Assembled Monolayers on Au Surfaces

Self-assembled monolayers are molecular assemblies that form spontaneously upon immersion of an appropriate substrate into a solution of the concerned molecule.31 The self-assembly method can provide highly ordered films without the need for complex equipment. Stoddart and colleagues synthesized bistable rotaxanes R3 with disulfide-based anchoring groups (Figure 3a) in order to allow selfassembly of the rotaxanes onto gold surfaces, and demonstrated the switching property of R3 by an electrochemical method.<sup>19</sup> The cyclic voltammetry (CV) of the SAM of R3 was recorded at a scan rate of 300 mV s<sup>-1</sup> at 288 K. CV is commonly the application of a cycle of voltage to a working electrode in solution. The measured



Figure 3. (a) Structural formula of a thiol-tethered bistable [2]rotaxane **R3** self-assembled on a Au surface. (b) The green cyclic voltammetry trace shows the starting-state **R3**, and the red cyclic voltammetry trace displays the presence of the switched-state molecule at a low temperature. (c) Schematic of the electrochemical switching of **R3** in self-assembled monolayers on a Au surface.

current at the working electrode can be an indicator of the oxidation-reduction reactions in solution. The first CV cycle (Figure 3b, green traces) displayed a high positive potential (+490 mV, compared with +290 mV for the dumbbell) on account of the TTF/ring charge-transfer interaction in the starting-state R3. The second cycle and all subsequent CV cycles (Figure 3b, red traces) did not display the +490 mV peak. Instead, a peak corresponding to the first oxidation (+280 mV) of the free TTF unit appeared in the CV. These observations provide direct evidence of the metastable (switched) state during the second and subsequent CV cycles. The first oxidation of the TTF unit in the ground-state molecule induced the CBPQT4+ ring to move (Figure 3c) to the DNP unit, generating a bare TTF unit that was captured in subsequent cycles at a lower temperature and a higher scan rate. The switched-state R3 reverted to its starting state by sweeping the CV cycle through an anodic potential (-600 mV), which reduced the CBPQT<sup>4+</sup> ring to the CBPQT<sup> $2 \cdot /2 +$ </sup> diradical.

#### **Polymer Matrices**

Stoddart, Heath, and colleagues demonstrated that TTF/DNP-based bistable [2]rotaxanes are switchable in another physical environment as well: the polymer matrix.<sup>22</sup> It is well known that in solution, the ground state of the TTF/DNP-based bistable rotaxanes displays an emerald green color for the  $\rm TTF/CBPQT^{4+}$  charge-transfer complex, while the metastable state possesses a ruby red color because of the DNP/ CBPQT<sup>4+</sup> charge-transfer interaction. When the bistable [2]rotaxanes R4 (Figure 4a) were admixed with an optically transparent polymer matrix, it was observed that the ground-state and the metastablestate molecules displayed different colors. Initially, the polymer film containing R4 displayed the green color of the ground state (Figure 4b); this turned to the reddish color of the metastable state at an applied potential of +1.0 V. The green color of the ground state was obtained again by removing the bias and allowing the molecules to thermally relax back to their ground state.

### Artificial Molecular Machines at Work

Nature demonstrates most elegantly that simple molecular mechanical components, once organized and assembled in a precise manner, can link motions efficiently from the nanometer scale to the macroscopic world. Linking these components also allows complex functions such as the powering of skeletal muscles, the synthesis of ATP (adenosine triphosphate), and the production of DNA and RNA. For example, the contraction and extension of natural muscles, the "best of the best" among the existing actuators, takes place by mutual sliding between myosin fibers (polymer of biomotors) and actin filaments (helical polymers of globular actin).<sup>32,33</sup> When appropriately oriented and organized, collections of these myosin/actin nanocomposite materials can give rise to substantial macroscopic movements in human muscle (Figure 5).

Inspired by nature, several research groups have been making efforts in utilizing the molecular machines' controllable mechanical motions, with an aim to develop engineering systems that operate with the same elegance, efficiency, and complexity as biological motors in the human body. Although this is still a lofty goal today, some impressive progress has been made along this exciting research direction in recent years. Different groups have proven that once organized, various forms of nanomachines can be used to manipulate much bigger objects (e.g., microcantilever beams, submillimeter-sized glass rods, and microliter droplets), mimicking what biomotors have achieved in human muscles.

Huang and colleagues have demonstrated a molecular-machine-based mechanical actuator by utilizing a hybrid top-down/bottom-up manufacturing approach.15,34,35 Here, the concept of a bistable [2]rotaxane was extended into a doubly bistable palindromic [3]rotaxane R5 to develop a molecular muscle (Figure 6a). R5 is composed of a symmetrical dumbbell component with two rings interlocked onto the dumbbell. The distance between two rings contracts and extends upon oxidation and reduction. Each ring carries a disulfide tether to allow the self-assembly onto gold surfaces of R5 through its two rings.

These molecular muscles, when selfassembled on microcantilever beams (500  $\mu m \times 100 \ \mu m \times 1 \ \mu m$ ), were shown to be capable of bending and stretching the beams when appropriate redox reagents were injected into the environment, a microfluidic cell (Figure 6b). Conversely, microcantilever beams that were coated with a redox-active but mechanically inert control compound did not display the same bending characteristics, as inferred from the relatively flat curves shown in Figure 6b. Heat, photothermal effects, and pH variation, as potential mechanisms for the observed curvature in the microcantilever beams, were precluded by conducting a



Figure 4. The electrochromic responses of a bistable [2]rotaxane **R4** in a polymer matrix. (a) Structural formula of **R4**. (b) Ground-state **R4** in a polymer matrix (labeled as the ground-state co-conformer, GSCC). (c) Metastable-state **R4** in a polymer matrix (labeled as the metastable-state co-conformer, MSCC). The three-electrode microfabricated device is used to electrochemically switch the states.

Myosin filament

Figure 5. The sliding of myosin (a biomotor) along actin filaments causes muscle contraction and extension.

series of control experiments. Moreover, using beam theory and analysis, it was shown that these observations support the idea that cumulative nanoscale movements within surface-bound "molecular muscles" can be harnessed to perform larger-scale mechanical work.<sup>34</sup>

Another dramatic illustration of the power of molecular machines was completed by Eelkema and colleagues.<sup>36</sup> They

proved that light-driven, unidirectional, molecular rotary motors R6 embedded in a liquid-crystal film are capable of rotating a glass rod that is ~10,000 times larger than R6. This molecule (Figure 7a) is composed of a rotary part (a right-handed helical structure), an axle (a central carboncarbon double bond), and a stator part. Upon doping of a nonpolymeric liquidcrystal film with R6, the helical organization induced by **R6** results in a polygonal fingerprint texture in the surface of the liquid-crystal film. When this R6-doped sample is irradiated with UV light of wavelength 365 nm, photochemical isomerization around the central carboncarbon bond on R6 occurs, which leads to the inversion of the helicity from righthanded to left-handed and the reorganization of the liquid-crystal film in a rotational (clockwise) fashion. Removing the UV light source causes a rotation in the opposite direction (counterclockwise). The rotation of the texture in the R6-doped liquid-crystal film can be utilized to manipulate a glass rod (5  $\mu$ m × 28  $\mu$ m) placed on top of the surface (Figure 7b). In other words, changes in the shape of R6 induce a remarkable rotational reorganization of the liquid-crystal film in which R6



Figure 6. (a) Schematic diagram of the bending of microcantilever beams by the self-assembled monolayers of palindromic bistable [3]rotaxane **R5**, and (b) the experimental data associated with the process.

is embedded and cause the rotation of submillimeter-sized particles on the surface.

In the third example, Berná and colleagues demonstrated the use of a molecular-machine-based photoresponsive surface to control macroscopic liquid transport across surfaces.37 In this work, a SAM of 11-mercaptoundeconoic acid (11-MUA) was assembled onto a gold surface, and photoactive rotaxanes R7 were physisorbed onto the SAM with the dumbbell of R7 parallel to the gold surface (Figure 8). R7 is composed of a ring and a dumbbell with a fluoroalkane station and a photoresponsive fumaramide station, which has a high binding affinity for the ring. Photoisomerization results in a transformation of the fumaramide into maleamide, which has a much lower affinity for the ring component. Thus, irradiation of light reduces the binding affinity to the ring drastically, and as a result, the equilibrium position of the ring is in favor of the fluoroalkane station instead of the fumaramide station. Therefore, the ring movement can be used to expose or conceal the fluoroalkane station, thus changing the surface energy. When small drops of low-volatility liquids (e.g.,  $CH_2I_2$ ) are deposited onto the molecularmachine-based photoresponsive surface, the collective operation of the R7 monolayer was sufficient to power the movement of a microliter droplet up a 12°



Figure 7. (a) Chemical structure of **R6**. (b) Rotation of a glass rod on an **R6**-doped liquid-crystal film. Pictures were taken at (left to right) 0 s, 15 s, 30 s, and 45 s, and show clockwise rotations of  $0^{\circ}$ , 28°, 141°, and 226°, respectively. Scale bars, 50  $\mu$ m.



Figure 8. (a) Schematic illustration of light-switchable rotaxanes **R7** physisorbed onto a self-assembled monolayer of 11-mercaptoundeconoic acid on Au(111). (b) Illumination with 240–400 nm light causes the rings to shuttle to the fluoroalkane units, leaving a more polarophilic surface.



Figure 9. (a)–(d) Macroscopic transport of liquids by surface-bound molecular machines **R7**. The dotted vertical line indicates the reference of motion in each image.

incline (Figure 9). In this experiment, ~50% efficiency was achieved; approximately 50% of the light energy absorbed by **R7** was used to overcome the effect of gravity.

#### Conclusion

When stimulated by light, electricity, or chemical reagents, mechanically interlocked molecules known as bistable rotaxanes-which are composed of mutually recognizing and intercommunicating ring- and dumbbell-shaped componentsexperience relative internal motions of their components just like the moving parts of macroscopic machines. The ability of bistable rotaxanes to precisely and cooperatively control mechanical motions at the molecular level has inspired researchers to develop molecularmachine-based engineering systems that operate with the same elegance, efficiency, and complexity as biological motors within the human body.

In past years, researchers have made tremendous progress along this line. They have proven that bistable rotaxanes are mechanically switchable in various solidphysical environments state (i.e., Langmuir-Blodgett films, self-assembled monolayers, and polymer matrices). Furthermore, different groups have demonstrated that cumulative nanoscale movements within surface-bound molecular machines can be harnessed to perform larger-scale mechanical work (e.g., driving microcantilever beams, rotating submillimeter-sized glass rods, and manipulating microliter droplets). By harnessing the nanoscale mechanical motion from artificial molecular machines and eliciting a nanomechanical response in larger-scale systems, these designs mimic natural skeletal muscle.

Although challenges remain, with collaborative efforts from experts in multiple disciplines such as chemistry, physics, and engineering, the comprehensive design, understanding, and application of molecular machine-based electroactive/photoactive polymers may become possible.

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