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Communication

The dynamic covalent reaction based on diselenide-containing crown ether irradiated by visible light

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ABSTRACT

A novel diselenide-containing crown ether (**BC7Se$_2$**) was fabricated, which can polymerize to form cyclic oligomers through intermolecular dynamic covalent reaction by irradiation of visible light. The size and distribution of oligomers are related to the monomer concentration. The decomposition reaction of oligomers is controlled by topology and solvents. Furthermore, potassium cation can inhibit the polymerization of **BC7Se$_2$** and accelerate the decomposition of oligomers.

Dynamic covalent bonds (DCBs) have attracted considerable attention and have a wide range of applications in dynamic combinatorial chemistry, stimuli-responsive systems and supramolecular chemistry owing to its specific reversible feature that DCBs can be cleaved and reformed under certain conditions [1]. Examples of DCBs including disulfide bonds, imine bonds, boronic ester and so on have successfully provided convenient methods to fabricate various 2D macrocycles [2] and 3D molecular cages [3] by external stimuli. The dynamic bond reaction can be tuned by various changes of reaction conditions such as temperature [4], light [1a,5], pH [1c,6] and guest molecules [7]. Compared to other stimuli, light is an outstanding stimulus with features of accurate controllability and friendly environment and has extensively used to manipulate the dynamic process [5a,8]. Disulfide bond-containing molecules can be triggered by irradiation of UV light to undergo dynamic covalent reaction. Compared with disulfide bonds (~240 kJ/mol), diselenide bonds with lower bond energy (170 kJ/mol) show more robust activity under much milder conditions [1a,5c,9]. And it has been demonstrated that metathesis reaction of diselenide bonds can take place by irradiation of visible light and stopped under dark [1a]. Dynamic covalent reaction based on diselenide and S-Se by the irradiation of visible light has been employed to construct polymer, responsive material [10]. However, to the best to our knowledge, the fabrication of macrocycles based on dynamic covalent containing-selenium bonds under irradiation of light is rarely reported [2a].

Macrocycles have extensive applications in many scientific communities. Especially, the discovery of crown ethers plays a seminal role in the development of modern supramolecular chemistry [11]. During these decades, a significant number of artificial macrocyclic structures such as cucurbiturils [12], calixarenes [13], cyclophanes [14] and pillararenes [15] have been developed. Macrocyclic compounds served as a type of crucial supramolecular host and have been extensively used in molecular recognitions [11d,12c,15d,16], molecular machines [17], smart materials [13b,18] and so on. Motivated by this, the fabrication of new macrocycles with novel structures and functions has become an essential goal in the supramolecular field. The introduction of various functional units or elements into the skeleton of macrocyclic compounds is one important strategy to obtain novel functional macrocycles. However, rational and efficient synthesis methods remain very rare, especially for the preparation of the macrocycle itself containing visible light-sensitive DCB.

In this work, we introduce a diselenide bond to substitute an oxygen atom in traditional oxacrown ether and present the synthesis of this novel diselenide-containing crown ether **BC7Se$_2$** using the potassium ion as a template. It is found that **BC7Se$_2$** can take place metathesis reaction to form cyclic oligomers (Scheme 1)

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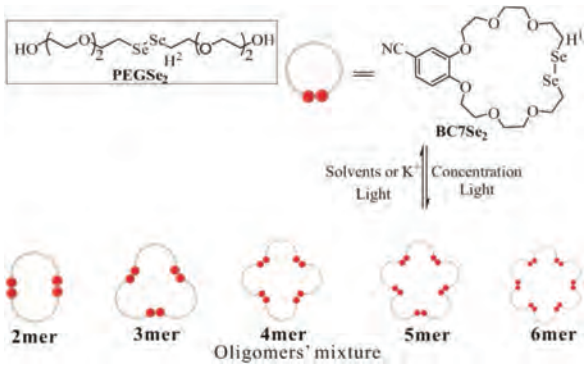

Scheme 1. Metathesis reaction between monomer **BC7Se$_2$** and its oligomers irradiated by visible light. Insert is the structure of **PEGSe$_2$** as control.

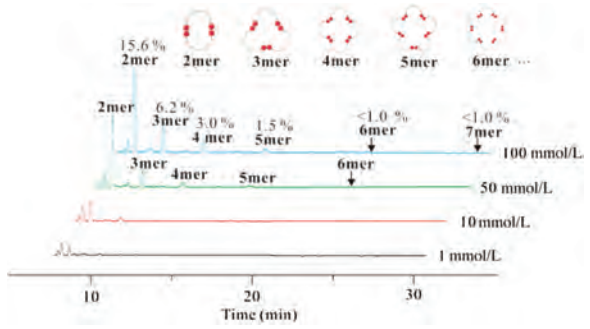

Fig. 1. Metathesis reaction of **BC7Se$_2$** irradiated by visible light after 36 h depending on concentration determined by HPLC. The peak for **BC7Se$_2$** is omitted for clarity.

in solution through the dynamic covalent exchange of intermolecular diselenide bonds under visible light without any other adducts and the concentration of **BC7Se$_2$** predominate the size of oligomers in this process. The decomposition reaction of oligomers is controlled by topological structures and solvents. Furthermore, potassium can inhibit the polymerization of **BC7Se$_2$** and promote the depolymerization of oligomers.

According to conventional approaches to fabricate oxacrown ether, the cyclization of **BC7Se$_2$** was carried out in the presence of potassium cation, which served as a template to induce cyclization reaction shielded from light (Scheme S1 in Supporting information). **BC7Se$_2$** was purified by silica chromatography column protection from light and obtained as a yellow solid. Interestingly, the ^{1}H NMR spectrum showed there were more than two components in the $CDCl_3$ solution. As shown in Fig. S12 (Supporting information), impurity signals not only in the aromatic region (7.15, 7.00 and 6.95 ppm) but also in the upfield (3.88–3.85 and 3.75–3.71 ppm), which were adjacent to OCH_2 of **BC7Se$_2$**, were considered to be the oligomers of **BC7Se$_2$**. The thin layer chromatography also verified **BC7Se$_2$** is the predominant component of the isolated product (Fig. S13 in Supporting information). The isolated product was dissolved in refluxing ethyl acetylate and *n*-heptane was dropwise added. Then the solution was cooled to precipitate relatively pure monomer **BC7Se$_2$** shielded from light.

A solution of **BC7Se$_2$** with a concentration of 5 mmol/L in CD_3CN was prepared and stirred under light generated by two incandescent lamps (220 V, 18 W) at room temperature for 36 h to monitor the dynamic covalent reaction based on **BC7Se$_2$**. LC–MS spectroscopy was used to determine the structure of the product (Fig. S14a in Supporting information). The sample was diluted to 0.1 mM to characterize by LC–MS coupled with HPLC. There were five molecular ion peaks for dimer of **BC7Se$_2$** at *m*/*z* 1066.0663 ($[M_2+NH_4]^+$), 1086.9945 ($[M_2+K]^+$), trimer of **BC7Se$_2$** at *m*/*z* 1589.0842 ($[M_3+NH_4]^+$), 1610.0133 ($[M_3+K]^+$), tetramer of **BC7Se$_2$** at *m*/*z* 1066.0679 ($[M_4+2NH_4]^{2+}$), 1075.5322 ($[M_4+K+NH_4]^{2+}$), pentamer of **BC7Se$_2$** at *m*/*z* 1323.5780 ($[M_5+2NH_4]^{2+}$), 1336.5422 ($[M_5+K+NH_4]^{2+}$) and hexamer of **BC7Se$_2$** at *m*/*z* 1588.0867 ($[M_6+2NH_4]^{2+}$), 1598.5504 ($[M_6+K+NH_4]^{2+}$) (Figs. S14b–g in Supporting information) excepted the ion signal of monomer **BC7Se$_2$**. The HPLC spectroscopy showed the dimer of **BC7Se$_2$** dominated the main component among these oligomers and the content of dimer increased to 4.5% from 1.5% (Fig. S15 in Supporting information). These observations confirmed that diselenide metathesis has occurred. However, it is difficult to distinguish the size of these macrocyclic structures on ^{1}H NMR spectroscopy due to the similar chemical environment of the proton (Figs. S12 and S16 in Supporting information). When the exchange reaction of **BC7Se$_2$** at 5 mmol/L was prolonged to 70 h under light, the contents of each macrocyclic molecules determined by LC–MS had no significant increase in HPLC and the reaction reached a balance. Therefore, the results motivated us to explore whether the exchange reaction correlated with the concentration of **BC7Se$_2$**. Samples with different concentrations of **BC7Se$_2$** ranging from 1 mmol/L to 100 mmol/L were prepared and exposed to visible light for 36 h, then were further performed on HPLC to test the dynamic combinatorial library (DCL) components (Fig. 1). The content of each oligomer in DCL was increasing with increasing the original concentration of **BC7Se$_2$**, and dimer remained kept the highest ratio among oligomers. Take dimer for instance, its content in the DCL was increasing to 15.6%, with the concentration of **BC7Se$_2$** increasing to 100 mmol/L. Surprisingly, the new heptamer of **BC7Se$_2$** ion peak at *m*/*z* 1850.1088 ($[M_7 + 2NH_4]^{2+}$) was first found on LC–MS spectroscopy at 100 mmol/L (Fig. S17 in Supporting information). Furthermore, the content distribution of each oligomer in DCL was decreasing, with the macrocyclic structure expanded. Interestingly, no linear oligomers were checked owing to the cyclic structure of the monomer. These observations firmly confirmed that the exchange reaction and the size of oligomers highly depend on the concentration of **BC7Se$_2$**.

A previous study [1a] has confirmed that noncyclic diselenide compounds could undergo diselenide metathesis under visible light and 50% of each reactant took place exchange reaction at equilibrium. However, the exchange reaction of **BC7Se$_2$** is weaker than diselenide-containing noncyclic molecules. It is considered that the difference between these two types of structures in dynamic covalent reaction was attributed to the topological structure. So, commercially available **BnSe$_2$** was used as a probe to test the exchange reaction between probe and **BC7Se$_2$** and noncyclic **PEGSe$_2$** that was designed to use as a control compound (Scheme 1), respectively. The metathesis reaction between **BnSe$_2$** and **PEGSe$_2$** has reached equilibrium under irradiation with visible light over 36 h and about 50% of **PEGSe$_2$** reacted with **BnSe$_2$** to generate a mixture containing the two reactants and the exchange product in a ratio of 1:1:2 (**PEGSe$_2$**:**BnSe$_2$**:product) determined by H^2 integration of **PEGSe$_2$** on ^{1}H NMR spectroscopy (Fig. S18 in Supporting information), which is consistent with the literature [1a]. On the contrary, only about 15% of **BC7Se$_2$** determined by H^1 integration of **BC7Se$_2$** on ^{1}H NMR spectroscopy took part in metathesis reaction with **BnSe$_2$** to yield a noncyclic structure containing diselenide under the same conditions (Fig. 2). These results suggested that the Se radical generated from **BC7Se$_2$** irradiated by visible light preferred to keep cyclic structure owing to the cyclic topology, rather than reacted with Se radical from **BnSe$_2$**. On the other hand, Se radical generated from noncyclic **PEGSe$_2$** and **BnSe$_2$**, respectively, could freely react with each other with the same chance.

Moreover, the reversible feature of dynamic covalent motivated us to study how to control the cyclic oligomers to depolymerize through dynamic covalent reaction. Solvents have important

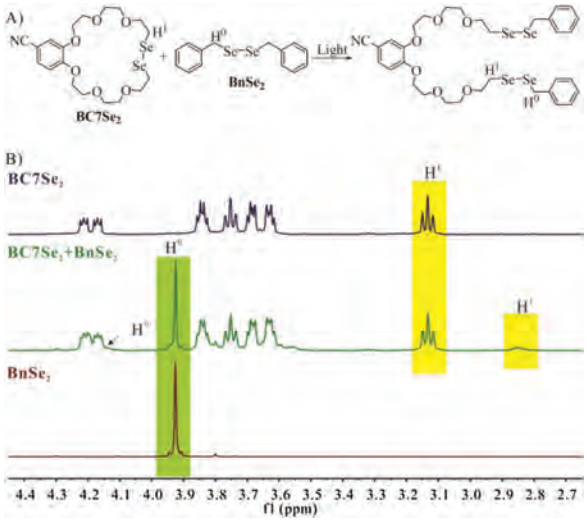

Fig. 2. (A) Metathesis reaction between **BC7Se$_2$** and **BnSe$_2$**. (B) Partial ^{1}H NMR spectrum of exchange reaction between **BC7Se$_2$** and **BnSe$_2$** irradiated by visible light after 36 h, [**BC7Se$_2$**] = [**BnSe$_2$**] =10 mmol/L (400 MHz, CD_3CN, 298 K).

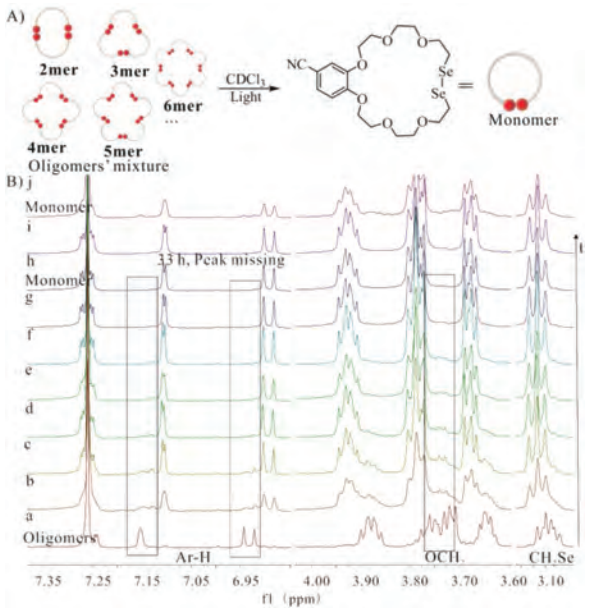

Fig. 3. (A) The schematic plot for the decomposition of oligomers. (B) Changes in ^{1}H NMR of oligomers based on **BC7Se$_2$** under light. (a) 0 h; (b) 1 h; (c) 2 h; (d) 4 h; (e) 6 h; (f) 8 h; (g) 22 h; (h) 33 h; (i) 55 h; (j) monomer of **BC7Se$_2$**. [Oligomers] = [**BC7Se$_2$**] =5 mmol/L, $CDCl_3$, 400 MHz, 298 K. The peaks in the rectangle represent partial proton of oligomers showed obvious chemical shift changes.

effects on organic reaction [19]. Thus, solvents effects and guest template were taken into account to investigate the reverse polymerization process of **BC7Se$_2$**. The mixture with a high content of cyclic oligomers was prepared by concentrating the **BC7Se$_2$** solution in dichloromethane by a rotatory evaporator under light due to the polymerization of **BC7Se$_2$** was under the control of its concentration. After drying under vacuum, the residue coated on the flask slowly switched from an oily shape to an elastic lump with yellow, which is different from the yellow powder of monomer **BC7Se$_2$** (Fig. S20 in Supporting information). A series of the solution containing oligomers of **BC7Se$_2$** with a concentration of 5 mmol/L calculated by the molecular weight of **BC7Se$_2$** was prepared in $CDCl_3$, $CDCl_3/CD_3CN$ = 1/2 (v/v), CD_3CN, DMSO-d_6 respectively under dark, the polarity of which range from low to high. Unfortunately, the oligomers of **BC7Se$_2$** could not dissolve in CD_3CN. The changes for these four samples under light were monitored by ^{1}H NMR spectroscopy. Before exposed to light, the Ar-H chemical shift of oligomers in the $CDCl_3$ (Fig. 3B–a) was located downfield compared with monomer **BC7Se$_2$** (Fig. 3B–j). On the contrary, partial peaks of OCH_2 of oligomers mixture shifted upfield compared to monomer. And the signal for the proton of monomer **BC7Se$_2$** is very weak in Fig. 3B–a. These observations reconfirmed the polymerization of dynamic covalent reaction for this system is under control of concentration. While the solution of oligomers in $CDCl_3$ was kept under dark for 4 h, there were no significant changes in the ^{1}H NMR spectrum (Fig. S19 in Supporting information), however, after exposed exposure to light, oligomers began to depolymerize to form monomer **BC7Se$_2$** and took about 33 h to be entirely consumed in $CDCl_3$ (Fig. 3) deduced from it is scarce to observed the ^{1}H NMR peak (in the rectangle) for oligomers (Fig. 3B–i). The results suggested that the depolymerization is dependent on light irradiation. And it took almost 194 h to complete this switch in the mixture solvents ($CDCl_3/CD_3CN$ = 1/2, v/v) (Fig. S21 in Supporting information). In DMSO-d_6 with higher polarity, the content of the oligomers and monomer **BC7Se$_2$** seemed to reach a balance, and the depolymerization reaction of oligomers stopped owing to there is no changes on the ^{1}H NMR spectrum after the sample exposed to light for 33 h (Fig. S22 in Supporting information). Although there were no apparent signals for oligomers on its ^{1}H NMR spectrum in CD_3CN before exposure to light, which was due to its weak solubility, there were more than two sets of chemical shifts after under light for 8 h (Fig. S23f in Supporting information). One set was attributed to monomer **BC7Se$_2$**. The other was considered as oligomers with small size due to the weak solubility of oligomers and the decomposition rate become slow after exposure to light for 55 h (Fig. S23i in Supporting information). These phenomena demonstrate solvent effect plays a crucial role in the depolymerization reaction.

In addition, K^+ template effect was used to induce the depolymerization of oligomers in the mixture solvents ($CDCl_3/CD_3CN$ = 1/2, v/v). In the presence of K^+ (3.0 eq.), it is calculated that oligomers took 148 h (Fig. S24k in Supporting information) to depolymerize completely, which is shorter than the case without K^+ template (194 h) determined by new peaks of monomer **BC7Se$_2$** substituted the signal of oligomers in ^{1}H NMR spectrum (Fig. S21). On the other hand, these two samples were concentrated under light, and the residue was resolved in $CDCl_3/CD_3CN$ = 1/2 (v/v) under dark to tested by ^{1}H NMR spectroscopy. The spectrum suggested the residue of the sample in the absence of K^+ is oligomers (Fig. S25 in Supporting information), and it can repeat depolymerization under light. Interestingly the residue in the presence of K^+ still kept monomer form **BC7Se$_2$** (Fig. S26 in Supporting information). All these results demonstrated that K^+ template could accelerate the decomposition of oligomers based on **BC7Se$_2$** and strongly inhibit the polymerization of monomer **BC7Se$_2$**.

In conclusion, we have described the synthesis of diselenide-containing crown **BC7Se$_2$** and its dynamic covalent reaction under the irradiation with visible light. To our surprise, **BC7Se$_2$** would rather form oligomers with cyclic structure through dynamic covalent reaction than occurred linear polymerization, and this process was controlled by topological structure and concentration of **BC7Se$_2$**. However, the depolymerization of oligomers was in the charge of solvents and potassium cation has template effects to enhance this process. Furthermore, the template effect of potassium ion can inhibit monomer **BC7Se$_2$** from forming oligomers through metathesis reaction. To date, one method for the decomposition of polymers to reuse monomer under mild conditions is to develop an efficient catalyst, which faced great challenges, otherwise, the development of dynamic covalent offers

an opportunity for the design of reusable polymeric monomer [20]. Therefore, the current polymerization and decomposition of **BC7Se$_2$** under light would be of particular significance, which may extend the research realm of diselenide bond beyond playing an important role in smart materials.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.cclet.2020.11.043.

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