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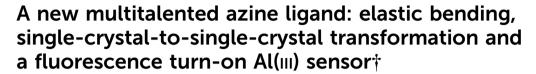


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We report a rare combination of two unique properties of an azine based ligand (H_3L): in a solid-state crystalline material it shows highly flexible and elastic behavior which on triggering with light results in slight deviation with phase transformation at the Single-Crystal-to-Single-Crystal (SCSC) level. Furthermore, in the solution state it acts as a highly selective, sensitive and reversible Al^{3+} sensor with a detection limit of 42 nM.

The design and synthesis of multi-tasking materials has attracted tremendous interest owing to their fascinating applications and economic viability. Although individual properties of the material in the solid-state and the solution state have been explored enormously, reports on combined studies comprising properties in both the solid-state and in solution are sparse. In the solid-state, crystalline materials are of paramount importance due to their potential applications in mechanical actuators,² phototransistors,^{3,4} light-emitting diodes (LEDs),⁵ solar cells,⁶ photonics and flexible electronics^{7,8} etc. In addition, crystal engineering is a powerful tool that helps organize complex materials into well-ordered crystalline lattices via the selfassembly of functional molecules. In fact, the engineered crystals are dominant for supramolecular materials, drug formulation, 10 artificial mechanosensors¹¹ and smart nanomaterials.¹² In crystal engineering, the major phenomena which attract attention are (i) the development of organic co-crystals (multi-component system), a prime focal point of pharmaceutical industries, (ii) single-crystalto-single-crystal (SCSC) transformations which involve several bond breaking and formation processes that lead to unusual products which may not be easily feasible by routine synthetic methods and currently (iii) investigation of the bending properties

of crystals as introduced by Desiraju *et al.* in hexachlorobenzene and further extensively explored by Naumov and Reddy *et al.* regarding their mechanical properties.^{13,14} All these features in crystal engineering are of high interest in the scientific community today.

Recently, mechanically flexible materials have gained much attention due to their promising application in pharmaceutical processes from formulation to tableting. 15 Generally, the flexibility of organic crystals depends on the mechanical force applied, which in turn depends on strong or weak interactions between lattice molecules. Most of the existing reports on bendable crystals are results of serendipity but lately Reddy et al. have reported a strategy for the design and synthesis of bendable crystals.8 So far, the reports on elasticity and plasticity of molecular crystals have been revolving around the following synthons: polyhalogenated N-benzylideneanilines, 16 organic co-crystals, 10 ester spacer based molecules, 15 naphthalene diimide derivatives, 8 Schiff bases, 16 vanillin derivatives8 and pyrimidine.17 However, to the best of our knowledge, so far, no report is available on azine based elastic crystals. Due to the presence of a flexible -C=N-N=C- azine unit, it is found that these crystals exhibit efficient internal stress dissipation, which is important for many applications involving flexible substrates. 18

The recent upsurge in the field of SCSC transformation reveals that transformed newer materials via external stimuli like light, heat and vapor have significant applications in sensor technology and gas storage. 19 The reversible/irreversible SCSC transformations in MOF and metal complexes are well documented.²⁰ However, in organic ligands, the SCSC transformations are rare and mostly restricted to [2+2] cycloaddition of two neighboring olefinic bonds which are designed to be close to each other within the range of 3.6–5.00 Å via introduction of a photo-induced strategy which has been extensively studied by MacGillivray and Vittal et al.21 To the best of our knowledge only one report is available on light induced SCSC transformation of non-olefinic ligands.²² Nevertheless, in the solution state, the relatively free movement of molecules makes it convenient to explore a huge repository of properties. In particular, organic luminous and non-luminous molecules play an important role in optics, electronics, materials and biosciences.²³ The recent developments related to the solution state have witnessed an

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Scheme 1 Synthesis of H₃L.

enormous increase in the research areas of aggregation induced emission (AIE) and ion sensing.24-26

Herein, we attempt to explore a unique combination of both the crystalline solid-state properties that deals with the bending properties of crystals which undergo SCSC transformation via light stimuli and further the solution state properties involving fluorescence turn ON for Al³⁺ sensing.

A new multifunctional azine based ligand (H₃L) was obtained by the reaction of synthesized (E)-3-(hydrazonomethyl)benzene-1,2-diol with 2-hydroxy-1-napthaldehyde (1:1) in methanol at room temperature for 15 h (Scheme 1). H₃L has been characterized by NMR and ESI-MS and was further authenticated by single crystal X-ray studies. The TGA study indicates a two-step rapid decomposition of H₃L at around 320 °C and 630 °C, while DSC studies show two phase transitions at 197 °C and 315 °C (Fig. S1-S5, ESI†).

 H_3L crystallizes in the monoclinic $P2_1/n$ space group (Fig. S6 and Table S1, ESI†). The crystal structure of H₃L reveals that the naphthyl group and benzene 1,2 diol units are separated by an E/Zaldazine unit which is arranged in the same plane. Molecular crystals of H₃L were grown in different solvents such as methanol, ethanol and acetonitrile, and in all these solvents the crystals of H₃L were found to be elastically bendable in nature. A systematic evaluation of the bending characteristics of the crystal was carried out, based on methods previously described by Ghosh and Reddy.²⁷ This was demonstrated by simply bending the crystal from both ends by using tweezers, and on release the crystal goes back to its original position (ESI,† Video 1).

In order to confirm the elasticity of the H₃L crystal, with much effort we mounted the crystal in a bent position by entrapping the crystal in a Y-shaped designed glass capillary and recorded the diffraction patterns by employing SCXRD techniques. Despite being bent we were able to index the unit cell parameters, which turned out to be the same as that of a normal crystal (Table S2, ESI†). We observed a slight decay in the intensities of the diffraction spots in the bent crystal as compared to the normal crystal, which is attributed to modified absorption and reflection characteristics of the crystals due to a change in the effective thickness of the crystal at the critical bending points during elastic bending or elongation²⁸ (Fig. S7, ESI†). However, we could not solve the structure with this quality of very weak diffraction. This confirms that even at the bent position the molecules are still intact in the lattice, thus retaining the crystallinity. Furthermore, on relaxation the same crystal shows the original unit cell. This provides a strong indication for a significant elastic stretching property of the crystal (ESI,† Videos 2-4). The different elastic stages of the successive bending in the H₃L crystal are shown in Fig. 1.

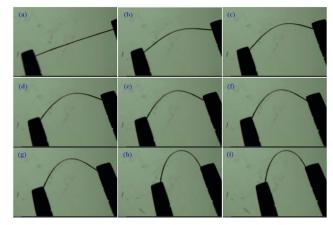


Fig. 1 The different elastic stages in the successive bending (a-i) in H_3L .

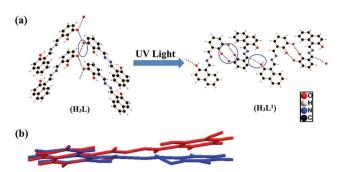
Bending experiments have been performed on the H₃L crystal. A model has been assumed wherein elastic bending introduces strain energy (E_s) due to compression and stretching of outer and inner arcs, respectively. Interestingly, the thinner crystals can be bent to a greater extent compared to thicker crystals due to the fact that the thicker crystals experience a greater level of strain energy, as the magnitude of change in Δl (where l represents the length of the crystal) for the inner and outer arcs will be greater for the same extent of bending.

The E_s will also be greater with a decrease in the critical radius, as can be seen from continuum mechanics.29,30 A simple calculation was performed to find out the values of Δl and critical radius r_c for the H_3L . The results of the calculations are explained in-depth in the ESI,† while a schematic of the bending experiment along with the type of bending experienced by the crystal is represented in Fig. S8 (ESI†).

The average change in translation along the 1D axis (Δl) is found to be 2.6743 Å per molecule for the H₃L crystal of 430 μm in length (0.43 mm). The calculation shows that the critical radius for breakage of the crystal, r_c , was found to be 30.8 μ m and the breaking strain is experienced on the crystal when the bending angular rotation per molecule is 0.0006134° per molecule. The calculations for the bent and original crystals show only minimal changes, during or after bending. This serves as further proof for the earlier indications of significant and exceptional elasticity of H₂L. The elastic nature of H₂L may be due to the tensile force between the naphthyl and benzene diol groups through an E/Z aldazine (-C=N-N=C-) unit followed by the attractive force from the van der Waals interactions.31

An earlier report by Barrett et al. 32 demonstrating light driven bending of azobenzene crystals prompted us to explore the SCSC transformation of the H₃L crystal under the influence of light. In this regard, the crystal of H₃L was exposed to UV irradiation, which resulted in the phase transformed crystal structure (H₃L¹). This SCSC transformation involved a change in the space group from $P2_1/n$ in H_3L to C2/c in H_3L^1 with approximately twice the length of the original a-axis (Table S1, ESI \dagger). The overlay molecular structure of H_3L^1 shows deviation of 11.77° from the H₃L plane (Fig. 2a and b).

The packing features of H₃L and H₃L¹ show the presence of intra and intermolecular H-bonding interactions. The intramolecular ChemComm Communication

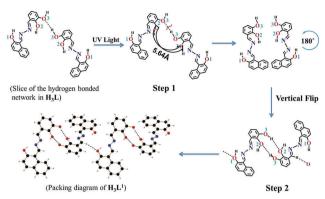


(a) Packing diagram of H_3L and H_3L^1 . (b) Molecular overlay structure of H_3L (blue) and H_3L^1 (red).

O-H···N interactions involve both the N atoms of an azine unit and the H atoms of hydroxy groups of the naphthyl and diol unit (Fig. S9, ESI†). The intermolecular O-H···O interaction in H₃L involves an H-atom of the diol group and an O atom of the adjacent diol group, with an O(3)-H(103)···O(3) bond length of 1.998(1) Å, leading to the formation of a dimer with the single hand shake position. Furthermore, each dimeric unit is connected to the neighbouring dimer forming a 1D-polymeric chain (Fig. S10 and Table S3, ESI \dagger). However, in H_3L^1 the intramolecular interactions are between the O and H atoms of the diol group from one molecule and the H and O atoms of the adjacent diol group $(O(3)-H(103)\cdots O(2), 2.106(9)$ Å) forming a dimer with the hand in hand handshake position with additional C-H···O, H-bonding interaction between an O atom of the napthyl unit and a hydrogen atom of the neighboring molecule $(C(12)-H(12)\cdots O(1), 2.602(13) \text{ Å})$, leading to the formation of a 1D-polymeric chain (Fig. S11 and Table S3, ESI†). This H-bonded 1D-polymeric chain structure suggests that relaxation processes are dominant which is favorable for elastic bending.33 Moreover, other azine based ligands L1 and L2 in the absence of -OH groups do not undergo photo-induced SCSC transformation (Schemes S1 and S2, ESI†). It is observed that the intramolecular H(101)···N(1) and H(2)···N(2) distances in H₃L are found to be 1.769(5) Å and 1.903(5) Å, respectively, whereas the similar intramolecular H(101)···N(1) and H(2)···N(2) distances in H₃L¹ are found to be enhanced considerably, 1.830(5) Å and 1.927(3) Å, respectively (Fig. S9, ESI†).

The SCSC transformation of H_3L to H_3L^1 can be explained by the probable mechanistic pathways as shown in Scheme 2. In the first step, on exposure of H₃L to UV light the O-H···O hydrogen bonding interaction is cleaved (step 1) which brings two adjacent molecules very close to each other from approximately 5.642 Å to 2.806 Å but due to strong hindrance between two bulkier naphthyl units, one of the molecules vertically flipped to 180°. Thus, the two adjacent molecules are strongly bonded to each other via strong O-H···O interactions (step 2) and subsequently each dimeric unit is stabilized by C-H···O interactions resulting in packing features of H₃L¹ as discussed above. Furthermore, the bending properties of the H_3L^1 crystal were also explored which displayed the same r_c value with enhanced elastic properties at a higher breaking strain of 0.001177° per molecule as compared to H₃L (ESI,† Video 5).

After displaying the excellent solid-state crystalline properties of H₃L and owing to its good solubility, we decided to investigate the



Scheme 2 Proposed pathway for the transformation of H₃L to H₃L¹

solution state properties. Aluminum is the third most abundant element on the earth's crust with a variety of applications. The development of highly selective and sensitive sensors for the detection of aluminum ions is of great importance in biological assays and environmental monitoring due to potential health implications caused by an overdose of aluminium.34-36

The photophysical behavior of H₃L was investigated via absorption and emission studies. The absorption spectra of H₃L in an acetonitrile (ACN) mixture (7:3 v/v in 10 μM HEPES buffer at pH = 7.2) was dominated by two absorption peaks at 325 and 382 nm wavelengths corresponding to the π - π * and $n-\pi^*$ transition, respectively. The subsequent addition of Al³⁺ in the form of Al(NO₃)₃·9H₂O results in a gradual decrease of the absorption maxima at 325 nm and a simultaneous increases at 382 nm with an isosbestic point at 344 nm. This provides strong evidence for enhanced binding of H₃L towards Al³⁺ ions (Fig. S12, ESI†). The emission spectra of H₃L exhibit a weak fluorescence peak at 500 nm. The titration of H_3L with Al^{3+} resulted in a gradual enhancement in fluorescence intensity at 555 nm with 50 nm bathochromic shift (Fig. 3a). A plausible reason for the fluorescence turn on towards Al3+ may be due to (i) the photo induced electron transfer (PET) suppressed by Al³⁺ coordination, (ii) the large bathochromic shift and (iii) the isosbestic point appearing in the absorption spectrum.³⁵ Thus, the results obtained from absorption and emission studies clearly indicate that the sensing behavior of H₃L is specific to Al³⁺ ions. The limit of detection (LOD) was determined to be 42 nM and the fluorescence response time was found to be 50 s (Fig. S13, ESI†).

The selectivity test was also performed towards various metal ions with concentration four times the concentration of Al³⁺. However, the fluorescence intensity is turned-on only in the presence of Al3+ ions with about 30 fold enhancement (Fig. S14b, ESI†); the results showed that other metal ions do not interfere with the intensity of the H₃L-Al³⁺ system and H₃L is specific for Al³⁺ (Fig. S14c, ESI†). Moreover, to understand the mode of complexation between H₃L and Al³⁺, ¹H NMR titration has been performed in DMSO-D₆ (H₃L) and D₂O (Al(NO₃)₃· $9H_2O$) solvents. On subsequent addition of Al^{3+} to H_3L solution, the -OH peaks at 12.87, 10.87 and 9.36 ppm decrease and finally at 0.8 equiv. of Al3+ all three -OH peaks completely disappear indicating the complex formation between H₃L and

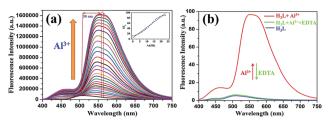


Fig. 3 (a) Fluorescence spectra of H_3L , inset: relative fluorescence intensity changes with respect to Al^{3+} concentration. (b) Fluorescence spectra of H_3L (20 μ M) in the presence of 20 equiv. of Al^{3+} before and after treatment with excess EDTA ($\lambda_{ex}=382$ nm).

Al³⁺ (Fig. S15, ESI†). The stoichiometry of $\mathbf{H}_3\mathbf{L}$ -Al³⁺ was confirmed by Job's method and the mass spectrum which shows $1:2(\mathbf{M}:\mathbf{L})$ complex formation (Fig. S16 and S17, ESI†). Furthermore, the reversibility of the chemosensor was also confirmed by the strong chelating agent ethylene-diamine-tetra-acetic acid (EDTA). The enhanced fluorescence intensity of $\mathbf{H}_3\mathbf{L}$ -Al³⁺ was quenched after adding EDTA, demonstrating that the binding of $\mathbf{H}_3\mathbf{L}$ with Al³⁺ is reversible in nature (Fig. 3b). The fluorescence quantum yield and average life time³⁷ of $\mathbf{H}_3\mathbf{L}$ -Al³⁺ was observed to be $\Phi = 0.182$ and $\langle \tau \rangle = 3.60$ ns (Fig. S18 and Table S4, ESI†). The performance comparison of $\mathbf{H}_3\mathbf{L}$ with other fluorescence sensors for Al³⁺ is shown in the ESI,† Table S5.

In summary, we successfully demonstrated an elastic crystal by introducing a new functionality such as an azine group. The elastic nature of the $\mathbf{H}_3\mathbf{L}$ crystals was found to be consistent in MeOH, EtOH and ACN solvents by producing long to very long length crystals. Photo-irradiation on $\mathbf{H}_3\mathbf{L}$ results in a SCSC phase transformation by twisting of the molecule along the a axis. Moreover, the mechanistic pathway of SCSC transformation of $\mathbf{H}_3\mathbf{L}$ to $\mathbf{H}_3\mathbf{L}^1$ has been explained. These findings in solid-state crystalline materials could be of vital importance in the field of crystal engineering and for the design of organic flexible materials, flexible electronic devices and optics. Furthermore, due to excellent solubility, the solution state sensing behavior of $\mathbf{H}_3\mathbf{L}$ was explored. $\mathbf{H}_3\mathbf{L}$ displays highly selective, sensitive and reversible sensing behavior towards \mathbf{Al}^{3+} with an LOD of 42 nM.

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Conflicts of interest

There are no conflicts to declare.

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