As an important research tool, peptide have received prominence in molecular biology, clinical research, instrumentation, study of protein structure and functioning, smart biomaterials etc. This has encouraged to develop synthetic peptides (by replacing enzyme sensitive amide linkage) i.e. Peptidomimetics having enhanced stability and attractive properties. 1,2,3-triazole motif is widely accepted isosteric group to surrogate such linkages present in biopolymers. Instead of using classical approach to peptide synthesis leading to poor yield, bad control over the stereo-and regioselectivity, solubility issues, slow reaction rate, use of toxic catalysts, difficult purification etc., we have used Topochemical Azide-Alkyne Cycloaddition (TAAC, lattice controlled) reaction. Utilizing the principle of crystal engineering, we have synthesized a structurally modified dipeptide (using alanine and valine) which undergoes heat induced topochemical 1,3-dipolar cycloaddition reaction to give triazole-linked pseudopolypeptide. Also, the crystals underwent cracking due to the packing strain generated at higher temperature due to thermal topochemical polymerization. Systematic characterization is carried out to study the following TAAC reaction.


**Keywords:** topochemical reaction, triazole, pseudopeptide