**Abstract:**

**Two new polymeric azo pigments of Cu(II) and Zn(II), [M(azopcyd)]n (where azopcyd = 4,4'-azodi(phenylcyanamide), have been synthesized and fully characterized. The azopcyd ligand was prepared in high yield from azodianiline by desulfurization reaction of its thiourea derivative. The polymeric azo pigments were prepared from the neutral azopcyd ligand and the aceteate salts of zinc and copper in acetone/water mixture. The presence of a sharp and intense band for the cyanamide stretching in the pigments provides evidence that both cyanamide moieties on the phenyl rings are equivalent. When the azopcyd coordinates to the metal ions, the ILCT band of the free ligand is still observed. The azo pigments can mix and melt with several polymers such as PVC and PE without any thermal degradation. The DSC/TGA data show that the azo pigments decompose at high temperature. The pigments are very stable in acidi, basic, oxidizing, and reducing media.**