ABSTRACT

Through an extensive study on the pyrolysis of coked raw rice husk (black ash) and subsequent to the incorporation of additives (silicon, iron, nickel, cobalt) as well as seeds (α-Si_3N_4), the conditions for the maximization of formation of single phase products (α-Si_3N_4, Si_2N_2O and β-SiC) are attempted to be identified. Attempts were first made to study the effect of coking temperature on the amount and nature of carbon, silica, C/SiO_2 molar ratio and changes in the additives present in the coked black ash. The detailed XRD and chemical analysis revealed that (a) with increasing coking temperature (>700°C), amorphous carbon tends to crystallise (b) volatiles loss is not constant till 900°C (c) presence of metallic additives suppresses the tendency towards crystallization of amorphous carbon at the coking temperature (d) metallic additives are present in amorphous compounds form in the black ash while nickel compounds tend to get partially reduced to elemental nickel.

The study on the formation of α-Si_3N_4 from untreated black ash through pyrolysis over a wide range of temperatures (1200-1450°C) revealed that (a) α-Si_3N_4 can be produced without any seed (b) No unreacted silica is left in the product at 1450°C (c) formation of Si_2N_2O can be completely suppressed. Addition of α-Si_3N_4 seed in different proportions resulted in the (a) increased formation of

α-Si_3N_4 (b) complete suppression of Si_2N_2O (c) increased conversion to α-Si_3N_4 at lower temperatures.

The study on the pyrolysis of black ash - silicon mixtures in the temperature range of 1150-1450°C revealed that (a) β-SiC whiskers begin to form at 1150°C (b) Si_2N_2O starts to form at 1200°C (c) β-SiC forms more rapidly as compared to Si_2N_2O (d) α-Si_3N_4 forms at 1350°C (e) α-Si_3N_4 formation is quite substantial by 1400°C.

The pyrolysis studies of Fe-, Ni- and Co-impregnated black ash revealed that (a) pure Si_2N_2O free from SiC, SiO_2 and Si_3N_4 can be produced at 1375°C (b) α-Si_3N_4 free from SiC and β-Si_3N_4 can be produced at 1365°C from iron impregnated black ash (c) cobalt and nickel additives favour the formation of β-Si_3N_4 (d) at lower temperatures (1250°C), Si_2N_2O is the dominant phase but at 1300°C, α-Si_3N_4 and β-Si_3N_4 both form and Si_2N_2O is completely suppressed (Ni, Co) (e) with increase in reaction temperature and time, content of β-Si_3N_4 drops (f) β-SiC does not form at higher levels of Ni and Co (in the starting black ash) in the temperature range of 1250-1375°C.

Key Words : Raw rice husks, Old rice husks, New rice husks, black ash, pyrolysis, Alpha - silicon nitride, Beta - silicon carbide, silicon oxynitride, Beta - silicon nitride, seed, additive, iron, nickel, cobalt.