CHAPTER II

MATERIALS AND METHODS

2.1 Materials

PVC : K-value = 66 (MW = 62500)

Filler : Calcium carbonate ($CaCO_3$)

Impact modifier : Methylmethacrylate butadiene styrene (MBS)

Stabilizers : -Tribasic lead sulphate

-Neutral lead stearate

-Calcium stearate

-Zinc stearate

-Epoxidized soybean oil (ESBO)

-Tris(nonylphenyl)phosphite

-Stearoylbenzoylmethane (SBM)

-Magnesium/aluminium hydroxide carbonate

-Butylated hydroxy toluene (BHT)

Lubricants : -Dicarboxylic acid ester

-Fatty acid ester

-High density polyethylene wax

-Lubricating pocessing aid acrylic polymer

2.2 Procedures

The raw materials were mixed in a high speed mixer (Papenmeier, model TGEHKV) in order to formulate the PVC compound in powder form (dry blend). This dry blend was milled in a two roll mill machine (Labtech, model LR150) to make a sheet and its heat stability was measured by static heat stability test (Oil immersion test : DIN 53381).

Each formulation has been tested for dynamic heat stability (Continuous rolling test) and then its surface appearance was measured by the application test (Extrusion test : Extruded in lab. extruder)

The formulation with the best heat stability and surface appearance was further characterized by its melt index (ASTM D 1238), izod impact strength (ASTM D 256) and vicat temperature (softening temperature: ASTM D 1525) to ensure that its other properties were also known. The plan is shown in Fig. 2.1.

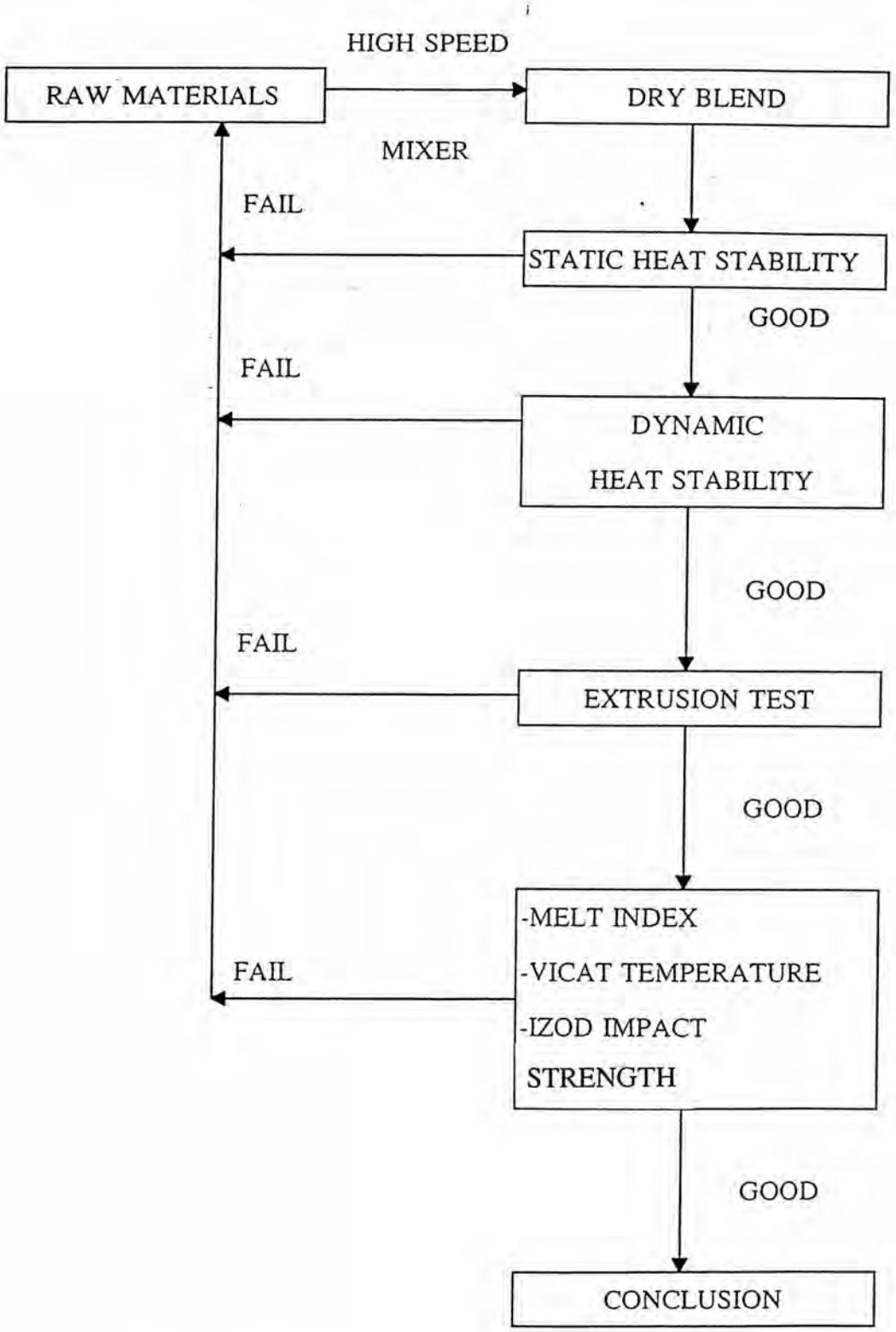

Fig. 2.1 : Experimental procedure

2.2.1 Physical characterization

2.2.1.1 Static heat stability test (oil immersion test DIN 53381)

2.2.1.2 Dynamic heat stability test (continuous rolling test) 250 g. of dry blend was first fused on a two-roll mill at constant temperature (195°C) and subjected to continuous stress on the same mill. Every 5-10 minutes samples were taken from the center of the sheet and used for the assessment of color change, if any.

2.2.1.3 Extrusion test

500 g. of dry blend was extruded by lab. extruder with a screw speed of 50 rpm. and temperature profile as follows:

Zone 1	Zone 2	Zone 3	Die 1
160°C	165°C	167°C	170°C

2.2.1.4 Melt index (ASTM D 1238)

2.2.1.5 Izod impact strength (ASTM D 256)

2.2.1.6 Vicat temperature (ASTM D 1525)

2.2.2 Experiments

2.2.2.1 The effect of stabilizers and co-stabilizers

In this part, Ca- and Zn-stearate were used as heat stabilizers with ESBO and tris(nonylphenyl)phosphite as co-stabilizers in

various amounts. (formulations are shown in Table A and B in the Appendix). The experiments were carried out in three phases.

First, the amount of Ca-and Zn-stearate were varied in the range of 3 to 8 phr. and 0 to 2 phr., respectively (formulations are shown in Table A in the Appendix). The dry blend samples obtained were assessed for static heat stability.

Second, to the optimized formulation from the first step with the randomly selected ratio of Ca- and Zn-stearate was added ESBO and tris(nonylphenyl)phosphite to study their effect as co-stabilizers on the heat stability and other properties. The amounts of ESBO and tris(nonylphenyl) phosphite were varied in the range of 3 to 7 phr. and 0 to 1 phr., respectively. (Table B in the Appendix)

Finally, to the optimum formulation from the second step was added stearoylbenzoylmethane (0.5 phr), and BHT (0.5 phr) to establish their effect on the heat stability acting as an inhibitor of the growth of polyene sequences and an antioxidant, respectively. Additionally, Mg/Al hydroxide carbonate was used as hydrogen chloride absorption stabilizer to study their effect on heat stability.

2.2.2.2 The effect of lubricants and the optimum formulation for heat stability and processability

The information obtained in section 2.2.2.1 was considered, and the formulation was adjusted to balance stabilizer and lubricant systems for good processability. The lubricant system was then adjusted in order to

avoid over-lubrication and plate-out problems by varying the amount of external and internal lubricants and the processing aid.