Change of Degradation Properties after Pelletizing of Soft Polyvinyl Chloride Dry Powder Blend by Melt Compounding

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Abstract

In current study, degradation properties of soft PVC dry powder blends were investigated before and after plasticating extrusion compounding by means of a torque rheometer as well as extent of discoloration. The powder blend was prepared thorough hot and cold mixing method. Before pelletizing by means of plasticating extrusion compounding, the PVC powder composite exhibited longer stability time. On the other hand, melt compounded pellets exhibited shorter stability and faster discoloration than that of powder blends. It was observed for the current PVC system that filling torque value is much higher for the granulated pellet samples than its dry blend form. The difference between discolorations increased as the heat duration increased during heat processing temperature value of 180 °C between dry powder blend and corresponding compounded pellets. It is concluded from the current work that pelletizing by melt compounding the studied PVC dry blends causes degradation changes in the PVC composites as it facilitates the dehydrochlorination process of the polymer. However, the PVC dry blend form has longer stability during PVC heat processing.

Keywords: Rheology, PVC, compounding, degradation, fusion, discoloration.

1. Introduction

Thermoplastic products manufactured by using polyvinyl chloride (PVC) as the raw material are one of the most used products in almost every part of our daily life. There are many types of these products such as window profile and pipes based on rigid PVC and, cables and hoses based on soft PVC materials. During manufacturing process, first; a powder blend which is a complex mixture of various ingredients such as stabilizers, plasticizers, fillers and pigments as well as PVC particles itself is prepared and then formed into various shapes either directly from the powder blend or indirectly from the pelletized form by means of a plasticating extruder.

PVC dry blends are used to provide unique properties to a wide range of end products. However, manufacturers are sometimes forced to convert these complex PVC dry powder blends into granulated pellet form (Muralisrinivasan, 2010) due to some advantages and also in order to use the recipes in other types of processing methods. In obtaining pelletized form of PVC dry blend, a pelletizing compounding extruder is used (Figure 1).



Figure 1: Schematic representation of the PVC pelletizing line, composing of a mixer and a melt compounding screw extruder.

Manufacturing from the pelletized form of PVC may have some advantages such as having homogeneity and integrity of PVC blend before shaping and also easier control over extruding process (Leadbitter, 1994).

There are arguments about how pelletizing affects the flow and fusion properties of the PVC composite material during processing since work done on the pellet form of the same dry blend powder material is not the same. It is the aim of this paper to observe and study experimentally the change of fusion properties after pelletizing of a soft PVC dry powder blend by melt compounding. In pelletizing process by melt compounding, the PVC resin is mixed with various ingredients in a jacketed mixing vessel during a heating and mixing cycle that is followed by a cooling cycle. Later, prepared dry blend is transferred into compounding extruder and pelletized at moderate temperatures by melt compounding. As a result, after die face cutting at the exit of the extruder, cooled pellets are packaged for the manufacturing of a demanded PVC product.

The data presented within this paper is a part of outcome of a project about optimization of PVC melts compounding. It is intended to prepare a base for evaluating a relationship for the fusion properties of dry blend and corresponding compounded PVC pellets at processing temperatures during degradation. For this purpose, rheological properties of both the dry blend and the prepared PVC pellets were measured by means of Thermo Haake Torque Rheometer (Thermo Fisher Scientific Inc., Germany). The color evolution was also followed by taking samples from the rheometer at every 10 minutes time interval.

2. Experimental Procedure

Experimental procedure implemented throughout this study is depicted in Figure 2. Two types of samples were prepared. First, a soft PVC powder blend formula was prepared by mixing the necessary materials. After cooling the PVC dry blend, samples were taken and noted as DB (dry blend sample). Later, prepared dry blend was melt-compounded and these pelletized samples taken were noted as GR (granulated pellets sample).

2.1. Preparation of Soft PVC Dry Blend

The formula used for the preparation of soft PVC dry blend is given in Table 1. Each ingredient was fed into the PVC mixer (DM-VD 300, Der-San Makina, and Turkey). The mixed powder blend was then dropped out into the cooling mixer at about 120 °C. The final PVC dry blend was discharged at 40 °C.



TORQUE RHEOMETER

Figure 2: Schematic representation of the experimental and testing set-up implemented throughout the study.

Table 1: Materials formulation used for the preparation of soft PVC powder blend.

| PVC (Norvinyl [®] S7060, K70) | 100.000 |
|---|---------|
| TiO_2 (Kronos [®] 1001) | 1.600 |
| CaCO ₃ (2 micron, Anadolu Mikronize, Turkey) | 84.000 |
| DOTP (SaSa, Turkey) | 64.000 |
| Stabilizer (Ca-Zn T42-153, Deva, Turkey) | 4.800 |
| Stearic acid (Akdeniz Kimya, Turkey) | 2,400 |
| Optical whitener (Dogan Ticaret, Turkey) | 0.016 |
| Ultramarine blue (Nubiola, Ferro [®]) | 0.032 |

2.2. Preparation of Pelletized PVC Powder Blend by Melt Compounding using a Twin Screw Extruder

A twin screw counter rotating parallel extruder with a diameter value of 110 mm was used for melt compounding (MCV 110-25D, Mikrosan, Turkey). The cooled PVC powder blend was fed into the extruder and pellets were obtained at the exit of the extruder with a die face cutter, after pellet cooling (Aydin, 2016).

2.3. Measurement of Fusion Properties and Colour Evolution

Fusion properties were measured using a Thermo Haake Torque Rheometer (Thermo Fisher Scientific Inc., Germany) equipped with a set of roller blade. During the test, roller blade rotation speed was kept constant at 50 rpm at a temperature value of 180 °C and torque versus time data were recorded by the Rheomix OS. This temperature value was chosen as being one of the processing temperatures for the extrusion manufacturing of soft PVC products. Discoloration was followed by taking samples from the rheometer at every 10 minutes time interval.

3. Results and Discussion

Results of fusion behaviour and discoloration for soft PVC dry blend sample (DB) and corresponding granulated pellet sample (GP) are presented together with their discussion below.

3.1. Evolution of Fusion Behaviour

The rheogram showing the variation of torque data with time is depicted in Figure 3 for both the DB and GP at the temperature value of 180 °C. It may be seen from Figure 3 that both samples show a typical behaviour of PVC composite compound in a mixer. After the initial filling peak value of $31.10 \text{ N} \cdot \text{m}$ for the sample GR, the torque curve drops to a minimum value of 2.80. Filling peak value for the sample DB which is in powder form remains at the torque value of $13.2 \text{ N} \cdot \text{m}$. After some time, PVC fusion takes place and equilibrium between shear heating and constant mixer temperature is reached, which results in a stable torque curve.



Figure 3: Rheogram showing torque value versus time for both the granulated pellet (GR) and powder dry blend (DB) samples at the temperature value of 180 °C.

On the other hand, the stable time for the sample DB is longer than sample GP. Hence, degradation of sample GR is expected to be in a shorter time than corresponding sample DB.

3.2. Evolution of Discoloration

The colour changes with time are depicted in Figure 4 for both the DB and GP samples at the temperature value of 180 °C. It may be seen from Figure 4 that for both the samples, discoloration is almost identical up to 80 minutes. After this time, sample GR begins to deform rapidly than sample DB. Chemical degradation begins earlier for sample GR than sample DB. There are so many parameters affecting the degradation process of the PVC composite system. It is observed from the current soft PVC formulation given in Table 1 that granulated pellet form of the same PVC dry blend exhibits shorter degradation process than its powder form.



Figure 4: Discoloration of the granulated pellet (GR) and powder dry blend (DB) samples at the temperature value of 180 °C and roller blade speed of 50 rpm during processing in the torque rheometer. Samples were taken throughout the mixing at every ten minutes.

4. Conclusion

Compounded pellets of soft PVC powder blend were prepared by melt compounding and degradation properties of both samples before and after extrusion compounding are detected by the extent of discoloration and the fusion rate of the degraded samples. It was observed for the current system that filling torque value is much higher for the granulated pellet samples than its dry blend form. This may be attributed to particle size of the pellets being greater than that of powder blend. The difference between discolorations increased as the heat duration increased during heat processing.

The difference for the stability time obtained from the measured rheogram was 10.30 minutes for the processing temperature value of 180 °C between dry powder blend and corresponding compounded pellets for the current PVC system. Although pelletizing of PVC is favoured by many manufacturers for some of the advantages but it is seen for the current studied system that pelletizing shortens the degradation time as observed from its rheological behaviour and discoloration during processing.

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