



EFFECT OF TEMPERATURE ON RHEOLOGICAL PROPERTIES OF SBR COMPOUNDS REINFORCED BY SOME INDUSTRIAL SCRAPS AS A FILLER

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ABSTRACT

The effect of fillers such as mix of carbon black and cement waste on rheological characteristics are studied in styrene butadiene rubber (SBR). The present study carried out through four recipes according to the loading level of mixed of industrial scraps (cement waste) and carbon black (N375) (C.B) that it used as reinforcement materials in the compounds. On the other hand, the rheological properties tests carried out by Rheometer according ASTM D 2705 specification in order to using these compounds in fender ship application. All compounds are composed of carbon black N375 (10, 20, 30, 35) pphr and cement waste (10, 20, 30, 35) pphr. The viscosity, minimum torque, maximum torque, scorch time, optimum cure time Tc90, optimum cure and thermoplasticity were examined at different temperature (165, 175, 185)°C. The results appeared that the rheographs were varied according to loading level, therefore; viscosity, minimum torque, maximum torque, optimum cure and thermoplasticity were decreased with increasing temperature and increased with increasing the loading level of mix C.B and cement waste. Also, the scorch time optimum cure time Tc90 was decreased with increasing temperature, loading ratio of C.B and cement waste.

Key words: Industrial scrap, Torque, Scorch time, Min. torque, Max. torque.

INTRODUCTION

Rheology is a study of deformation and flow, in a simplistic way the deformation may be associated with elasticity and the flow with viscosity. Then, rheology is synonymous to viscoelasticity. Indeed, sometimes these terms are used interchangeably the performance characteristic of a pharmaceutical suspension depends upon its rheological properties. An

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extensive treatment has already been given for the viscoelastic behavior of gum rubbers and compounds¹. In recent years, fiber-reinforced composite materials were frequently used as engineering materials in structural parts of marine field such as ships, harbour facilities and floating structures, fishing trawlers, domes of submarines because of favourable properties such as high strength to weight ratio, high modulus, chemical stability, fatigue resistance and ease of manufacturing. It was also shown that the reinforcement of flax fillers in the polypropylene increased the relative dielectric permittivity.

Rheograph is a cure curve obtained by a “Oscillating Disc Rheometer”, it is one instrument to determine the kinetic crosslinking. An oscillating rotor is surrounded by test compounds, which is enclosed in a heated chamber. The torque required oscillating the rotor and it is monitored as a function of time². From the cure curve of torque against cure time, all the vulcanization characteristics of the rubber compound can be determined directly. Rheograph is divided into 3 phases, which are explained in Fig. 1³:

- 1- Phase-1: It represent processing behavior of the rubber compound.
- 2- Phase-2: It describes the curing characteristics of the rubber compound.
- 3- Phase-3: It gives an indication of physical properties of the rubber compound.

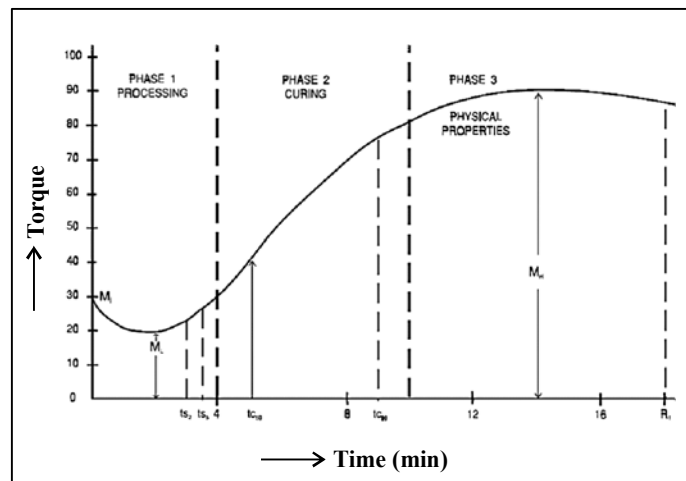


Fig. 1: Rheograph (cure curve)³

The plot of torque against time is analyzed to obtain the various results. In oscillating disc rheometer, the rheograph is displayed in real time and at the end of test time and computer analyses the graph, results are automatically computed and displayed on

the screen or the printer. The displayed results of rheograph are categorized into three reports^{4,5}:

- (i) Torque report in (Ib-in)
- (ii) Time reports in minutes
- (iii) Derived reports

1- Torque reports

ML (Minimum Torque)

As the compound gets heated under pressure, the viscosity decreases and the torque fall. The lowest value of torque recorded is called ML. Basically, it is a measure of the stiffness and viscosity of vulcanized compound, from these values of torque, the viscosity obtained by the relation:

$$= ML * 2.7 \quad \dots(1)$$

MH (Maximum torque)

As the curing starts, the torque increases proportionately. Depending upon the type of compound, the slope of rising torque varies. After a while, the torque typically attains maximum value and it plateaus out, it is called "plateau curve". If test is continued for sufficient time, the reversion of cure occurs and torque tends to fall. This type of curve with reversion is called "reverting curve". At times the torque shows continuous rising trend during the period of record. Such type of curve is called "rising or marching curve". MH (Max. torque) is the highest torque recorded in plateau curve. In reverting curve, the Max. torque recorded is abbreviated as MHR. Maximum torque can be expressed measure of stiffness of vulcanized rubber.

Time reports

Scorch time TS2

After attaining minimum torque, during cure phase, scorch time TS2 is recorded as the torque rises 2 units above ML, scorch is premature vulcanization in which the rubber becomes partly vulcanized before the product is in its final form and ready for vulcanization. It reduces plastic properties of the compound so that it can no longer be processed. Scorching is a result of both the raising of temperature during processing and the amount of time that compound is exposed to elevated temperatures. This period before vulcanization starts is generally referred to as "scorch time". Since scorching ruins the rubber, it is important that vulcanization does not start until this process is complete⁶.

Optimum cure time Tc90

It is the time at which 90% of cure has taken place⁷.

Reversion time RT

It is the time to reach 98% of MH after passing MH. It gives us an indication of the quality of the compound as to how long it retains its physical properties when subjected to heat ageing. Reversion occurs with over cure, in this case the modulus and tensile strength decrease.

Derived values

Cure rate index CR

Cure rate is an essentially a measure of the linear slope of the rising curve. The rate of cure is the rate at which cross-linking and the development of stiffness (Modulus) of the compound occur after the scorch point, when the compound is heated beyond the scorch point, the properties of the compound changes from a soft plastic to a tough elastic material required for use. During the curing phase crosslinks are connect the long polymer chains of the rubber together. As more crosslinks are formed, the polymer chains become more firmly connected and the stiffness (modulus) of the compound increases. The rate of cure is an important vulcanization parameter since it determines the required time for complete curing process. Cure rate index is simplified parameter can be given by the following equation⁴.

$$CR = 100/(Tc90-TS2) \quad \dots(2)$$

Difference between maximum and minimum torques ΔM

The difference between maximum and minimum torques can be expressed as a parameter of crosslink density. We used this value in the studies of crosslink density as indicator for evaluating the crosslink density.

EXPERIMENTAL

The materials which were used in this research are:

- **SBR** used in these experiments is SBR1502 contain 23.5% styrene content and butadiene. It has the specific gravity (0.95 g/cm³), supplied by the Petkim, Turkey.

- **Carbon black N375** supplied by Doudah, Iran. It is examined in accordance with the DBP absorption (ASTM D136) and Iodine absorption (ASTM D135).
- **Cement waste:** (Kiln dust) supplied by Karbala factory. The chemical analysis of cement waste powder shown in Table 1.

Table 1: Analysis of cement waste powder

| Materials | % |
|--------------------------------|-------|
| SiO ₂ | 11.11 |
| Al ₂ O ₃ | 2.38 |
| Fe ₂ O ₂ | 2.55 |
| CaO | 46.29 |
| MgO | 1.12 |
| SO ₃ | 0.59 |
| Cl | 0.12 |

Zinc oxide (97%) and stearic acid (99.4%) were supplied by Durham, U.K. 6PPD N-(1, 3-Dimethyl butyl)-N-Phenyl-Para-Phenylenediamine (98%) was supplied by Flexsys, Belgium. MBS N-oxydiethylenebenzothiazole 2-sulfonamide (98.2%) supplied by ITT, India. The South Patrol Company supplied Paraphenic wax, processing oil. Sulfur was supplied by Al-Meshrak CO. Iraq

Equipment

Laboratory mill: Baby mill was used in this study to prepare the batches. It has two roll mills, having provisions for passing cold water. These rolls are cylindrical in shape and of 150 mm diameter and 300 mm in length in the other hand the roll speed is 20 r.p.m.

Rheometer: The cure characteristics of the different compounds were measured at 165, 175, 185°C at time = 12 min, by MV-ODR- (Micro vision Enterprises-India) according ASTM D2705⁷. The optimal vulcanization time, scorch time and torques of the compounds were determined by this rheometer.

Preparation of recipes

The recipes were prepared by mill laboratory; the compounding ingredients are shown in Table 2.

Table 2: Ingredients of recipes

| Ingredients | Compound (1) | Compound (2) | Compound (3) | Compound (4) |
|----------------|--------------|--------------|--------------|--------------|
| SBR 1502 | 100 | 100 | 100 | 100 |
| Zinc oxid | 5 | 5 | 5 | 5 |
| Stearic acid | 2 | 2 | 2 | 2 |
| Paraphinic wax | 2 | 2 | 2 | 2 |
| Processing oil | 5 | 5 | 5 | 5 |
| Carbon black | 10 | 20 | 30 | 35 |
| Cement waste | 10 | 20 | 30 | 35 |
| 6PPD | 0.5 | 0.5 | 0.5 | 0.5 |
| MBS | 1 | 1 | 1 | 1 |
| Sulfur | 1.5 | 1.5 | 1.5 | 1.5 |

RESULTS AND DISCUSSION

The Figs. 2-3 represented all the variations in rheograph properties. Initially, there is suddenly increase in torque as the chamber is closed, as the rubber is heated, its viscosity decreases causing decrease in torque. Evenly, the rubber compound begins to vulcanize and transform to elastic solid and the torque rises. Molecular chain scission may be occurring.

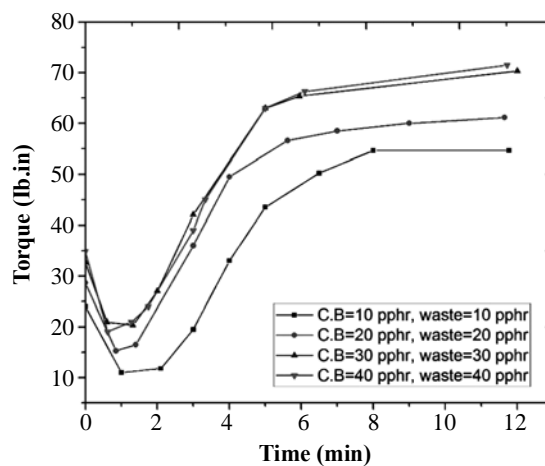


Fig. 2: Variations in rheograph of SBR compounds with the loading level of mix C.B. and waste at T = 165°C

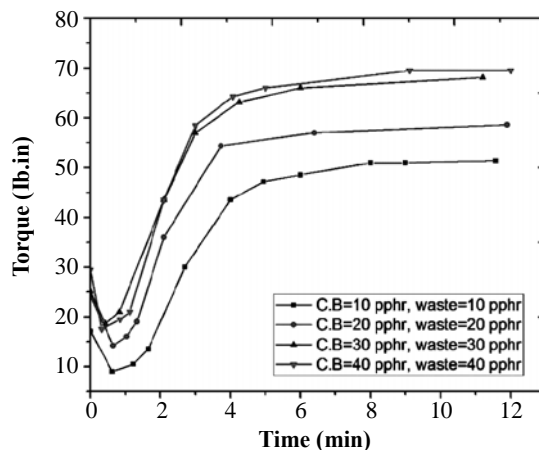


Fig. 3: Variations in rheograph of SBR compounds with the loading level of mix C.B. and waste at T = 175°C

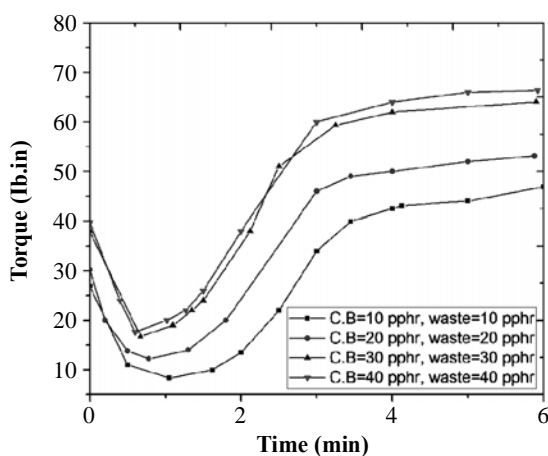


Fig. 4: Variations in rheograph of SBR compounds with the loading level of mix C.B. and waste at T = 185°C

However, an increasing torque indicated that cross linking is dominating, and then the torque reach plateau, this indicated that the completion of curing and formation of stable network^{8,9}. Therefore, min. torque, max. torque and the time values such as scorch time, and optimum cure time and the derived properties such as cure rate index, viscosity, and thermoplasticity. One can see from these graphs that the rheograph curves and properties values were varied with increase the loading level of mix of C.B. and cement waste at constant temperature at: T = 165°C for Fig. 2, T = 175°C for Fig. 3 and T = 185°C for Fig. 4, in the same time these values were varies with different temperature for the same loading level.

The Figs. 5-7 show the relation between min. torque, max. torque, viscosity against temperature, respectively. It is clear from the figures, that these properties were decreased with increasing temperature and increasing with increase loading level of mixed C.B, cement waste.

This behavior attributed to an increasing the cross linking density and correlations between the mix of C.B. cement waste and the rubber chain, this leads to increasing the torque and viscosity, and then leads to increase the vulcanization process. so the effect of temperature of these volcanzite was increasing the rate of vulcanization at short time.

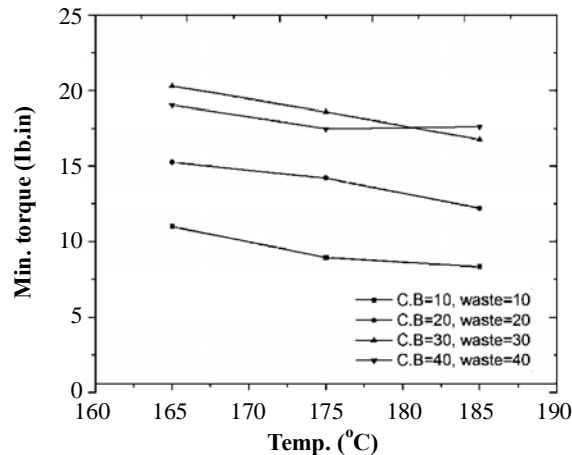


Fig. 5: Variation of minimum torque with temperature for different loading ratios

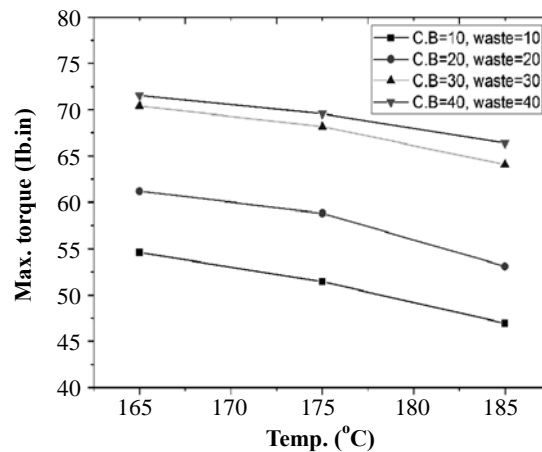


Fig. 6: Variation of maximum torque with temperature for different loading ratios

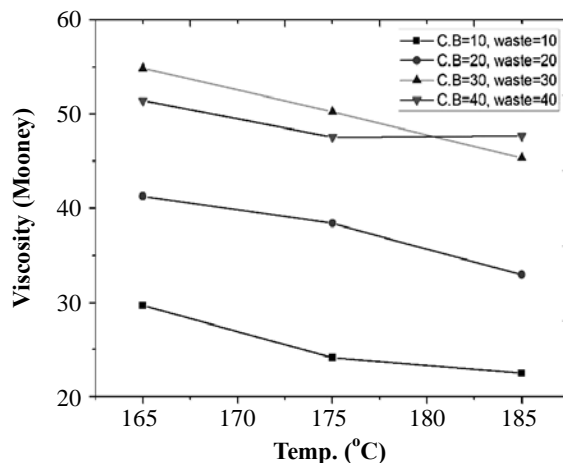


Fig. 7: Variation of viscosity with temperature for different loading ratios

Figs. 8-10 show that the relation between scorch time, optimum cure time Tc90, optimum cure against temperature. It is clear that these properties were decreased with increasing temperature and loading level of mixed C.B. cement waste. This behavior attributed to increasing the vulcanization rate and decreasing the values of Opt.cure time Tc90 and optimum cure.

Fig. 11 show the relation between thermoplasticity against temperature. It can be seen that this property was increased with increase temperature and decreased with increase loading level of mixed C.B. cement waste.

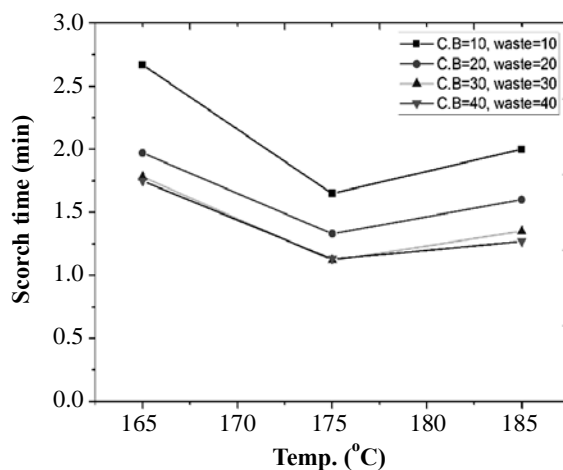


Fig. 8: Dependence of scorch time with temperature for different ratios

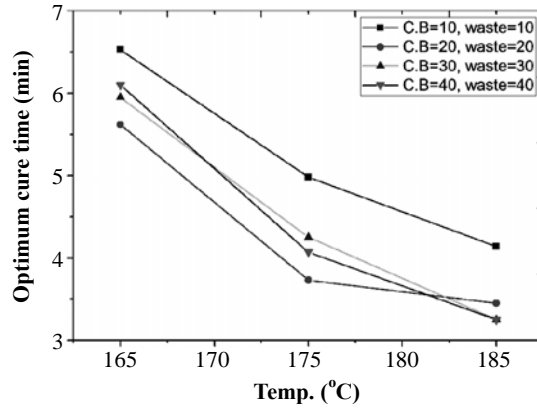


Fig. 9: Dependence of optimum cure time on temperature for different loading ratios

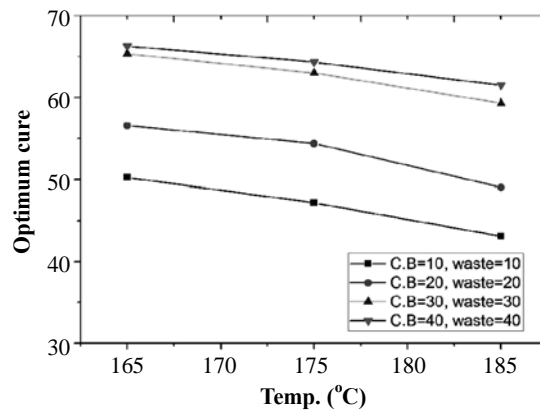


Fig. 10: Dependence of optimum cure on temperature for different loading ratios

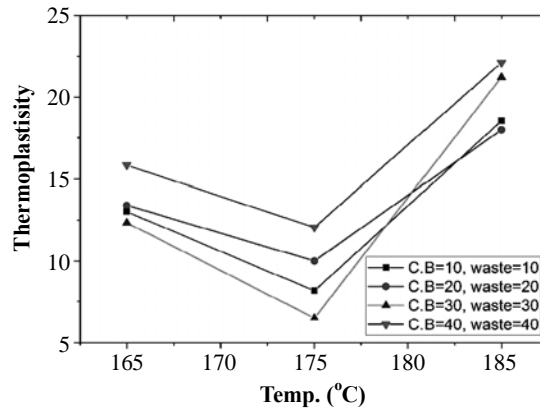


Fig. 11: Graphical relation between thermoplasticity and temperature for different loading ratios

CONCLUSION

- (i) All these properties, which were studied decreasing with increase temperature except thermoplasticity.
- (ii) Increasing of min. torque, max. torque, optimum cure, viscosity and thermoplasticity with increase loading level of mixed C.B. and cement waste.
- (iii) Scorch time, optimum cure time were decreased with increase in loading level of mixed C.B. and cement waste

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