Research on the fire endurance of the silica sand membrane

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Abstract. In this study, the fire endurance, density, low-temperature deflection, penetration and ring-ball method tests of the membrane produced throughout the mixing of Bitumen, IPP (isotactic polypropylene), LDPE (Low-density polyethylene) calcite and silica sand at varying proportions were performed and the test results were compared with the membrane used today. In our study, TS 120 EN 1427, TS 118 EN 1426, TS EN 13501-1, KG-TL-029 testing methods were used. The density of the silica material we have obtained was measured at 1.3782 g/cm³, the endurance of the material was found out to be better, softening temperature was measured at 155°C, brittleness temperature at -7°C and the fire class was found out to be as E.

1. Introduction
With the energy resources coming closer to depletion, the need for isolating the buildings from the inside or outside with the purpose of using the resources efficiently has emerged. As a result of thermal insulation, it is both possible to save energy and keep the internal climatic conditions at the desired level. When it comes to buildings, sections such as roofing is among the places where thermal insulation should be applied [1].

The primary external factors that the buildings are exposed to are water and moisture. Various waterproofing and drainage applications are carried out in building constructions in order to eliminate or minimize the effect of water and moisture [2]. Considering the fact that we spend most of their time in closed environments; one of the ways to provide a safe structure and a comfortable indoor environment is to ensure that the building elements surrounding the place are sufficiently dry. In order to achieve this, it is necessary to take all measures against water. Measures that are taken to remove the water from a structure prolong its life while also increasing its endurance against earthquakes [3].

The fire must be taken under control before it spreads in order to minimize property damage during events of fire. Spreading is slowed down by setting up of fire-resistant parts inside the building. The resistant parts made of fireproofing material restrict the spreading of fire for certain periods and provide the firefighting teams with sufficient time to intervene before the fire expands. Thus, the risk of property loss is minimized by fireproofing. In addition, fireproofing measures should be also taken for the exterior façade to prevent a fire from spreading across the floors and adjacent buildings and to avoid life and property loss. The inability of the firefighters to intervene due to an increased temperature resulting from the rapid expansion of fire in buildings which lack fireproofing may result in total damage of the building [4].

As an alternative to expensive and imported insulation materials, producing a mineral based, naturally structured, artificial fibre and boron mineral enforced fireproof insulation material by using completely local resources (such as limestone and silica sand) is necessary [5].
In this work, we have conducted is to meet the increasing energy need of our country and to reduce dependence on foreign countries by increasing the fire endurance of the insulation materials that are used with the awareness of the fact that the energy use and loss (heat, sound, water, etc.) is at its most in residential areas. This way, both the energy conservation will be ensured by the prevention of unnecessary energy loss and comfort will be provided to people by ensuring property and life safety with the increase in fire endurance. As a result of this energy conservation, contributions to the country economy will be made with the decrease in energy imports and energy dependence on foreign countries. In addition to that, the fire endurance effect will be determined throughout the production of silica sand membrane.

2. Material and method

2.1. Bitumen

The bitumen is defined as a viscous liquid or a solid substance which contains mainly hydrocarbons and its derivatives; it is soluble in trichloroethylene, non-volatile and gradually softening when heated [6].

Figure 1. Bitumen.

2.2 Isotactic Polypropylene (IPP)

Polypropylene, specifically isotactic polypropylene (IPP), the most common commercial thermoplastic polymer, has a significant power: weight ratio due to its low density and high endurance. Polypropylene crystals have a melting point of about 160°C and are usually processed over 200°C [7].

Figure 2. Isotactic Polypropylene.

2.3 Low-density polyethylene (LDPE)

LDPE is produced by the high-pressure process (at 1600 atm and 200°C) of polyethylene. LDPE, which has many branching in its molecular structure compared to HDPE, therefore has lower crystallinity. For this reason, its hardness and durability are lower, and chemical endurance is limited. In contrast, its impact resistance is higher. The density of LDPE which can be used between -40-70°C is between 0.91-0.92 g/cm³ [8].
2.4 Calcite
Calcite; chemical formula CaCO$_3$, is a mineral which is the constituent of limestone which has a crystal grain size between 1 mm-100 mm. According to the Mohs hardness scale, its hardness is 3, and its specific gravity is 2.7 g/cm$^3$ at 20°C [9].

2.5 Silica Sand
Silica sand belongs under the group named insulated lightweight building materials. Silica sand is composed of silica particles smaller than 2 mm that are formed as a result of the separation of granite-type rocks. Although the average surface area varies depending on the region that it is extracted, it is about between 100-200 m$^2$/g. It is used in the refractory industry, silica brick production, casting industry and glass industry [10]. The results of the analysis of silica sand are given in Table 1.
Table 1. Sand Analysis Report.

| Sieves | Oversize | Multiply | Percentage | Experiments | Acceptance | Criteria | Experiment |
|--------|----------|----------|------------|-------------|------------|----------|------------|
| 2.0    | 0        | 0        | 0.00       | Max         | +2         | Min      | Result     |
| 1.6    | 0        | 0        | 0.00       | AFS         | +2-2       | 87.35    |
| 1.0    | 0.00     | 0        | 0.00       | Moisture %  | 3          | 8        |
| 0.710  | 0.00     | 0        | 0.00       | Clay %      | 0.1        | 0.5      | 0.35       |
| 0.500  | 0.03     | 0.75     | 0.06       | SiO₂ %      | 98         | 99       | 98.10      |
| 0.355  | 0.28     | 9.8      | 0.56       | Fe₂O₃ %     | 0.16       | 0.40     | 0.16       |
| 0.250  | 1.36     | 61.2     | 2.73       | Al₂O₃ %     | 0.5        | 1.2      | 1.08       |
| 0.180  | 5.19     | 31.4     | 10.41      | Sintering % |            |          |            |
| 0.125  | 31.35    | 2539.35  | 62.86      | Temp (°C)   | 1500°C     | 1750°C   | >1500°C    |
| 0.090  | 10.69    | 1261.42  | 21.44      | Temp (°C)   | +4         | -4       |
| 0.063  | 0.85     | 139.4    | 1.70       | Loss on ignition % (LOI) | 0.02 |
| Pan    | 0.12     | 33       | 0.24       | Acid demand value (ml) |          |
| Total  | 49.87    | 4356.32  | 100        | Sample amount | Vibration duration | 6 minutes | x |
| AFS    | 87.35    | 50 g     | x          | x           |
| Avg. micron | 151.29 | 100 g    | 10 minutes |

2.6 Method
In this study, the fire (heat) endurance, density, low-temperature deflection, penetration and ring-ball method tests of the membrane produced throughout the mixing of Bitumen, IPP (isotactic polypropylene), LDPE (Low-density polyethylene) calcite and silica sand at varying proportions were performed and the test results were compared. In this study, TS 120 EN 1427, TS 118 EN 1426, TS EN 13501-1, KG-TL-029 testing methods were used.

2.7 Production of the material (membrane)
The bitumen was heated and mixed in a steel pot to 150°C until it became liquid (Figure 6a). Then the recycling oil at a rate of 2.8% was melted and mixed into the liquid bitumen (Figure 6b). After that, IPP was added at a rate of 2.75% to give our mixture hardness (Figure 6c). The mixture was melted up to approximately 190°C-200°C by adding 3% LDPE for viscosity. After the polymers melted, we added our filler materials (45% calcite + 5% silica sand) (Figure 6d) and melted it by mixing it at 190°C-200°C for 1 hour.
We poured the mixture into the cylindrical moulds for penetration testing and we put it on the steel tray for other tests and let it cool down.
The penetration test was performed in two different ways at temperatures of 25°C and 60°C. The prepared material was poured into cylinder moulds with the diameters and heights of 4 cm. After 15 minutes of cooling, the mixture was placed in water baths (Figure 7b). It was kept for 35-40 minutes until the temperature is fixed. The penetration measurement of the needle was performed on the penetration device (Figure 7c).

In order to measure the degree of the softening, a Ring-ball mechanism was installed with 2 pieces of balls with the weight of 3.5 grams (Figure 8). It was subjected to a heating test in a glycerine bath. The temperature was noted at the exact time the balls fell down and its softening degree was recorded.
2.10 Low-temperature deflection test
Our 13 cm x 5 cm rectangular shaped and 3 mm thickness sample was put under deflection in cold test in a Cold-Flex device.

2.11 Density test
The density test of our material was done with a pycnometer. After the sample was placed in the slot of the pycnometer, the lid was closed and the gas in the sample was emptied. Then our density test was completed and density values were determined from the pycnometer.

2.12 Fire endurance test
The samples that were prepared at 90 mm x 250 mm x 3 mm were tested with an E-class fire testing device (Figure 9).

![Figure 9. Fire testing device.](image)

3. Results and discussion
3.1 Penetration Test Findings
As a result of the test conducted on the penetration device for 5 seconds at 100 g, the penetration measurements of the needle were found at 18.3 mm, 14.1 mm, 11.7 mm at 25°C, 97.6 mm and 99 mm at 60°C respectively.

3.2 Ring and Ball Test Findings
We noted the temperature at the moment the balls fell down and we recorded the softening by measuring it at 155°C.

3.3 Low-temperature Deflection Test Findings
After holding our sample at -5°C in the device for an hour, we have observed that our sample was not broken. Then, we held the sample at 0°C for an hour and we again didn't observe any breaking. Then after putting our sample to rest for 30 minutes, we again didn't observe any breaking at -5°C for an hour. Then, after letting the sample rest for another 30 minutes, we have tested the sample at -7°C and we have observed that breaking occurred in the sample.

3.4 Density Test Findings
We measured the density from the pycnometer at 1.3782 g/cm³.

3.5 Fire Endurance Test Findings
As a result of the test, no big difference was observed between the normal production (50% calcite) and the sample (5% silica + 45% calcite) and its fire class was determined as fire class E.
4. Conclusion
The concluding observations are as follows:

• The results of the penetration tests done for 5 seconds at 25°C and 100 g are measured at 18.3 mm, 14.1 mm and 11.7 mm respectively. As for 60°C, while the material that is used in the marked is measured at 125 mm and 143 mm, the measurement values of the material we have produced is at 97.6 mm and 99 mm. According to the results, it can be said that our material is harder.

• According to ring and ball test that we have conducted to determine the softening degree, the temperature at the moment the balls fell down was recorded as 155°C for the material we have produced. According to ring and ball test that we have conducted to determine the softening degree, the temperature at the moment the balls fell down was recorded as 155°C for the material we have produced.

• According to the low-temperature deflection test, we have produced was broken at -7°C.

• From the pycnometer, we have produced was measured at 1.3782 g/cm³.

• As a result of the test, no big difference was observed between the normal production (50% calcite) and the sample (5% silica + 45% calcite) and its fire class was found out as E.

• It is observed that fire class remained as E.

When all the results are taken into consideration, there was no change in class for the silica material we tested compared to the equivalent materials used in the market, however, improvements were made in terms of density and endurance.

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