Radiant heat protective performance of clothing assemblies with flexible aerogel-Nomex nonwoven composite as thermal insulation

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In order to achieve a superior level of heat protection, aerogel-Nomex nonwoven composite felts have been prepared and used as middle layer in 3-layered fabric. To study the effect of precursor concentration on the radiant heat protection, methyltrimethoxysilane (MTMS) precursor to methanol molar ratios are varied for making the flexible aerogel-Nomex nonwoven felt. The heat protective performance of three-layered clothing assemblies, with aerogel-Nomex nonwoven felt as the thermal insulating middle layer [outer layer Nomex IIIA woven and inner layer modacrylic-cotton (60:40) woven fabric] has been determined by exposing the clothing combinations to pure radiant heat fluxes by using Stoll's criterion. The use of aerogel-Nomex nonwoven fabric shows more than 100% increase in the heat protection time as compared to the conventional multilayered heat protective clothing assembly. The aerogel-Nomex nonwoven fabric samples produced using MTMS precursor are found to be soft and flexible as compared to the conventionally produced pure silica aerogels, by using tetra-alkoxysilanes, which are normally fragile in nature.

Keywords: Aerogel-Nomex nonwoven, Composites, Methyltrimethoxysilane, Multilayered protective clothing, Nomex fibre, Nonwovens, Radiant heat flux

1 Introduction

It is well known that people often confront extreme hot environment during firefighting, where they may be exposed to high intensity radiant heat fluxes or flames or both. They can receive burn injuries in such situations, and that may be even fatal for them. The firefighters' protective clothing is a multicomponent clothing assembly, which is primarily designed to protect a person involved from various types of thermal hazards. Apart from providing adequate protection from heat, fire and some chemical hazards, clothing used for such purposes must be light, adequately durable, and comfortable in normal working environment and must provide convenience during interventions.

Traditionally, wool and cotton or rubberized cotton tunics were being used by firefighters since the beginning of professional firefighting¹. Natural fibres, viz. wool and cotton can be given flame resistant finishing treatment to make them flame resistant and can be used alone or in blends. Synthetic fibres, e.g. viscose, polyester, nylon and modacrylic can also be

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made flame retardant using flame retardant finishes or using additives prior to spinning^{2,3}. A number of heat resistant fibres, e.g. thermosets, *meta* and *para*aramids, polybenzimidazoles, polybenzoxazoles, and semicarbon have found their application in such clothing assemblies². These thermally-resistant organic polymeric fibres are having a certain degree of resistance to thermal and chemical degradations. These properties have been attributed to the chemically inert structures of the polymer, and fully aromatic or ladder like chain configurations⁴.

The thermal energy radiated from a source of heat is absorbed and transferred through the protective clothing materials. The heat energy subsequently passes through the clothing layers and get absorbed by the skin layers causing burn injuries. The heat flow through the clothing assembly is delayed by many complex mechanisms. Many factors, including the type of fibres being chosen, effective thermal conductivity of clothing layers, bulk density and porosity, specific heat capacity, radiative properties, finishes being applied, extent of moisture, affect the rate of heat flow through the fabric.

Although various materials such as phase change materials (PCM) and shape memory alloys (SMA)⁵⁻⁷ have found application in superior thermal

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insulations, "aerogel" exhibits extreme thermal insulation properties. Aerogels were first produced and characterized by Kistler⁸, but its full potential as a useful material is yet to be explored fully. Aerogels, being of very low density and nano porous materials of extremely low thermal conductivity, is considered as a potential candidate for super thermal insulation. Kistler and Caldwell⁹ measured the thermal conductivity of silica aerogel granules of different sizes at different levels of air and mechanical pressures. The thermal conductivity of silica aerogel was found to be about 0.02 W m⁻¹ K⁻¹ at normal atmospheric condition, and about 0.01 W m⁻¹ K⁻¹ under very low vacuum conditions.

Aerogels may be transparent as well as opaque to visible light, have low dielectric constants, and have very low sound velocity (<100 m/s) (ref. 10). Instances of superior heat and fire protection have been found in literature, where aerogels incorporated lightweight clothing was used for heat protection^{11,12}. Mohanpriya *et al.*¹³ measured the thermal conductivity, thermal diffusivity, and evaporative resistance of aerogel particle loaded polyester/ polyethylene nonwoven composites. They concluded that the fibrous structural density, and the aerogel present in the fibre aerogel composite has significant effect on the thermal insulation properties.

Aerogels can be incorporated in clothing layers by using different methods. In which form and how different aerogels can be utilized to enhance the thermal protective performance of such clothing assembly, has not yet been explored extensively. From the application point of view, monolithic aerogels have the drawback that they cannot be handled easily or used simply to protect complex shaped objects¹⁴. They are delicate and fragile, having a problem of shedding dust. Therefore, the fibre reinforced aerogel fabric has been produced as an alternative to achieve a flexible, drapable and more durable thermal insulating material.

In the present study, in order to produce a useable flexible Nomex-aerogel nonwoven felt, methyltrimethoxysilane (MTMS) precursor has been chosen¹⁵⁻¹⁷. The aerogel has been directly formed into the Nomex nonwoven fabric by the formation of gel first in the fibrous material, and then dried by liquid CO_2 supercritical drying method to produce the aerogel-Nomex nonwoven felts. These aerogel-Nomex felts have later been characterized and evaluated for radiant heat protective performance. These felts were used as a middle layer in a three-layered fabric combination, with a Nomex-III woven outer layer and a modacrylic/cotton woven fabric as the inner layer. Such combinations are similar to those used for firefighters' protective clothing. The heat protective performance of these proposed three-layered clothing assemblies have been evaluated by exposing them to purely radiant heat flux of very high intensity (~35 kW/m²). The time taken to cause the second degree burn injury has been noted using the Stoll's second degree burn criterion¹⁸.

2 Materials and Methods

2.1 Materials

Nomex (*m*-aramid) fibres of 2.44 dtex (2.2 den) and length 50 mm were procured from the manufacturers and used for the preparation of nonwoven fabrics. Fabric was produced on Dilo laboratory model needle punching line (fabric areal density $150.0 \pm 5.0 \text{ g/m}^2$, 150 punches/cm² and depth of needle penetration 10 min). The chemicals used for the preparation of silica gels to be formed in the nonwoven fabrics were: methyltrimethoxysilane (MTMS, Fulka Comp. Switzerland), oxalic acid (COOH)₂. 2H₂O (Qualigenic Fine Chemicals, India), ammonium fluoride (NH₄F, Loba Chemie), and methanol (Merck, India). Double distilled water was used for the preparation of all the solutions. A Nomex-IIIA (Nomex 93%, Kevlar 5% and 2% antistatic fibre) woven fabric as an outer layer (OL), a modacrylic/cotton (60/40) woven fabric as an innermost layer (IL), and Nomex nonwoven felt (with and without embedded aerogel) were used as the middle layer (ML), in the three-layered protective clothing assembly. Heat protective performance of the novel three-layered clothing assemblies are compared with the conventional fabric assembly. Fabric assemblies are produced as follows:

Novel fabric assembly:

OL (Nomex woven) – ML (aerogel+Nomex nonwoven) – IL (modacrylic/cotton woven 60:40)

Conventional fabric assembly:

OL (Nomex woven) – ML (Nomex nonwoven) – IL (modacrylic/cotton woven 60:40)

2.2 Methods

2.2.1 Preparation of MTMS based Gel in Fibrous Structure

The methyltrimethoxysilane (MTMS) based silica alcogel was prepared in petri dishes in two steps (acid and base), following the proceedings as reported by Rao et al.¹⁶ (Scheme 1). To study the effect of variation in molar ratio of MeOH:MTMS (M), the solutions were produced in four different molar ratios of 15.4, 25.6, 35.9 and 46.2 respectively and these solutions were used to produce aerogel-Nomex felt samples with codes M4, M3, M2, and M1 respectively, as described in this section. Alcosols of different molar ratios were prepared in 150 mL beakers, by mixing MTMS, methanol and oxalic acid followed by stirring for 1 h. The resulting sols were allowed to stand for 24 h for hydrolysis and afterwards, the base catalyst NH_4F (ammonium fluoride, 5 M) was subsequently added drop by drop with continuous stirring for 15 min. After addition of the base catalyst, the sols were poured onto the *m*-aramid nonwoven fabric placed in petri dishes and allowed to form gel. After 8 h, the gel formation was found to be completed and the fibre-alcogel composites were transferred to liquid CO₂ supercritical drying (SCD) chamber.

The hydrolysis and condensation of MTMS take place according to the reaction shown in Scheme 2 (ref. 16):

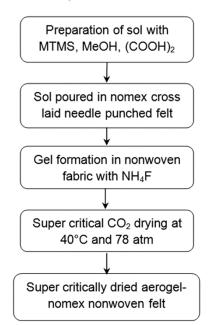
In the MTMS precursor, every monomer has one non-hydrolysable methyl group (-CH₃) and three hydrolysable methoxy (-OCH₃) groups. The three methoxy groups participate in the hydrolysis and subsequently in condensation reactions, whereas the methyl groups (-CH₃) are responsible for the super hydrophobicity. As the condensation reaction proceeds, the number of methyl group increases, creates discontinuity in the molecular network, and probably leads to relatively soft and flexible aerogel material, in contrast to the traditionally produced fragile aerogel products.

2.2.2 Supercritical CO₂ Drying of MTMS based Gel

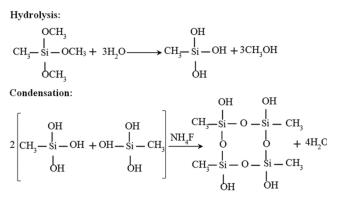
The supercritical liquid CO_2 drying chamber consists of a jacketed cylindrical space, where the coolant is circulated through the jacket (outer shell) to bring the inside temperature down to about 5°C, simultaneously inside pressure is adjusted to get liquid CO_2 . The fibre-alcogel composites are placed inside the inner chamber and then filled with the liquid CO_2 . The existing solvent (methanol) in the gel was first exchanged with the liquid CO_2 for several times till the pores of the gel were completely filled with the liquid CO_2 , then the gel was held at supercritical state (at 40°C and 78 atm pressure) for 2 h and subsequently the remaining CO_2 was drained out in that state only. At the supercritical state, no liquid-gas boundary exists, and hence there is no surface tension. The capillary force is the reason for the shrinkage and collapse of the solid gel matrix in the ordinary drying. With no surface tension, the capillary forces are absent in such condition, and the structure of the solid gel matrix is retained. These aerogel-Nomex nonwoven composite felts were used as the middle layer for the evaluation of thermal protection, with Nomex IIIA and cotton/modacrylic woven fabric as outer and inner layers respectively.

2.2.3 Measurement of Fabric Parameters

All the fabric fundamental characteristics, e.g. thickness, fractional cover, areal density, and thermal conductivity were measured. The areal density or the mass per unit area (g/m^2) of all inner and outer layer



Scheme 1 — Sequence of preparation of MTMS based aerogel-Nomex nonwoven felt



Scheme 2 — Hydrolysis and condensation of methyltrimethoxysilane (MTMS)

fabrics were determined following the ASTM D3776 standard. The outer layer and inner layer fabric thicknesses were determined as per the ASTM D1777 standard, using a digital thickness tester, at compressional pressure of 2000Pa. Ends and picks per unit length of the woven fabric samples were determined following the ASTM D3775 standard, using pick glass. The areal density (g/m^2) and the thickness of the nonwoven fabric were determined following the ASTM D6242 and ASTM D5729 standards. All the samples were conditioned in standard atmosphere for 24 h before the measurements were taken. Some fundamental results of the tested individual fabric layers are shown in Table 1. The fabric bulk densities were determined from the measured areal density and the thickness of the fabrics and the aerogel composites using the following formula:

Bulk density
$$(kg / m^3) = \frac{\text{Areal density } (g/m^2)}{\text{Thickness } (mm)}$$
 ...(1)

2.2.4 Measurement of Thermal Resistance

The thermal resistance of all the fabric components of the clothing assemblies was determined using the Alambeta thermal conductivity measuring instrument, measures thermal properties which the of compressible textile materials, like woven or nonwoven fabrics. The instrument consists of two parallel circular plates. The fabric sample to be tested is placed on the bottom plate, which is at ambient temperature and the top plate heated to 10°C higher than the environmental temperature is lowered and brought in contact with fabric sample, which presses the fabric sample at a pressure of 200Pa. The measured heat flow values are used to calculate the thermal properties of the aerogel-fabric composites¹⁹. The measured thermal resistance values for all the fabric layers, including the aerogel incorporated nonwoven layers, were obtained, and given in Table 1.

2.2.5 Estimation of Protection Time

The estimation of protection time was done on an instrument, which was developed in our laboratory based on the guidelines given in ASTM F1939 standard. The radiant heat flux is created with a vertically oriented radiant heating source, consisting of five short wave (\sim 1.4 to 3 μ m) infrared heating tubes, 1,500 Watt each. The intensity of the radiant heating can be controlled by using a voltage variac. The clothing combinations, prepared with or without aerogel-fabric samples, were tested at an intense heat flux of $\sim 35.0 \text{ kW/m}^2$, for the evaluation of radiant heat protection. The clothing combinations were exposed to the radiant heat flux and the temperature rise on the other side of the fabric was measured using a blackened copper plate, attached with a resistance temperature detector (RTD) sensor. The temperature data was converted to cumulative thermal energy (J/cm^2) and plotted simultaneously with Stoll curve to find an approximate estimate of the second degree burn injury time on continuous heating. The following formulae have been used to calculate the heat flux and cumulative thermal energy:

Heat flux $(kW/m^2) =$

$$\frac{m \times Cp \times (T_{final} - T_{intial}) \times 10^{-3}}{[\text{Absoritivity} \times \text{area} \times (t_{final} - t_{intial})]} \quad \dots (2)$$

Cumulative thermal energy $(J/cm^2) = m \times Cp \times (T_{\text{final}} - T_{\text{initial}})/\text{ area of copper plate } \dots (3)$

where m(g) is the mass of the copper calorimeter; Cp (J g⁻¹ °C⁻¹), the specific heat capacity of copper; T_{final} and $T_{initial}$ (°C), the final and initial temperatures of the copper plate; absorptivity ~0.9; and t(s), the time. For the calculation of heat flux, the initial linear portion of the temperature curve was considered, as it has been suggested in standard ASTM F1939. After each test,

Fabric	Thickness, mm	Bulk density kg m ⁻³	Cover	Thermal resistance×10 ⁻³ m ² K W ⁻¹
Nomex IIIA woven (outer layer)	0.44	500.0	0.94	15.0
Nomex NW (middle layer in conventional fabric)	2.85	52.0		145.0
Modacrylic:cotton (60:40) (inner layer)	0.41	478.0	0.81	24.2
Aerogel+Nomex (middle layer in novel fabric, M1-M	4)			
M1	3.64	78.8		137
M2	4.04	101.5		145
M3	4.50	112.4		173
M4	4.52	122.1		188

some oily deposits due to the condensation of gaseous products evolved during pyrolysis, were found to appear on the blackened side of the copper plate and it was cleaned with a piece of clean cloth.

3 Results and Discussion

The microstructural features of the MTMS based aerogel-Nomex fabric felts have been observed using scanning electron microscope (SEM). The acquired SEM images are shown in Fig. 1. Figure 1(a) shows the image of aerogel-Nomex nonwoven composite felt, observed under ordinary microscope, and Fig. 1(b) shows an SEM image of the Nomex nonwoven fabric only. The air gaps of the open fibrous structure can be observed to be filled with nanoporous MTMS based aerogel [Fig. 1(a)]. The changes in the surface characteristics of the fibres due to formation of aerogel on its surface can be observed in Fig. 1(c). At higher magnifications (×30K) an approximate idea of the aerogel particle size can be noticed, which is about 600 nm [Fig. 1(d)]. The larger size particles that have been observed can further be composed of many smaller sub particles of aerogels in this case.

The FTIR studies of the aerogel-Nomex felts were done in the wave number range 600-4000 cm⁻¹, on Perkin Elmer (model. 760), using ATR- FTIR technique. Figure 2 shows the FTIR spectra of the MTMS based aerogel-Nomex nonwoven felt. The FTIR investigation of the aerogel-Nomex fabric shows characteristic peaks of silica based aerogels for Si-O-Si linkage of dense silica network (~1000 -1150 cm⁻¹, antisymmetric stretching vibration)²⁰. The presence of $Si-(CH_3)_3$ is confirmed from the absorption peaks at 1260 cm⁻¹ (due to symmetric bending vibration of C-H), along with peaks occurring at around 850 cm⁻¹ and 750 cm⁻¹. The peaks at around 2960 cm⁻¹ are due to symmetric stretching of C–H, and the peak observed at around 1460 cm⁻¹ is due to antisymmetric bending vibration of C-H at structural unit -CH₃. The small O-H vibrations at around 3500 cm⁻¹ and 1650 cm⁻¹ indicate the presence of residual free -OH groups and physically absorbed H₂O molecules¹⁷. Strong absorption peaks (800-850 cm⁻¹) due to Si–C can be observed for these aerogel– Nomex nonwoven composite (Fig. 2).

The presence of a large number of CH_3 groups makes the MTMS based aerogel-Nomex composite felts as super hydrophobic, thus meeting the requirement of stability, which is important from the application point of view. The hydrophobicity of the aerogel-Nomex nonwoven felt has been examined using a contact angle measuring instrument (Krüss, DSA 100), and the recorded contact angle is found to be in the range of ~145°–165°. In general the hydrophobic aerogels can be produced using the precursors and co-precursors of the type R_nSiX_{4-n} [R is the alkyl or aryl or vinyl groups, X is alkoxy groups, n = 0 - 3]¹⁶. In the present study, the trifunctional organosilane compound of the type R_JSiX_3 is selected, namely the methyltrimethoxysilane [MTMS, H₃C–Si–(OCH₃)₃]. Both the methyl and the methoxy groups are the smallest among all the alkyl and alkoxy groups that can therefore be expected to facilitate the hydrolysis and condensation

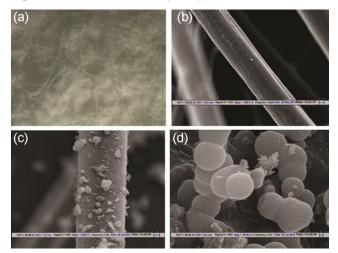


Fig. 1 — SEM images of aerogel-Nomex nonwoven composite fabric samples at different magnifications [(a) aerogel-Nomex nonwoven composite felts seen under optical microscope; (b) only Nomex nonwoven fabric at $\times 3K$; (c) aerogel-Nomex nonwoven fabric felt at $\times 5K$; and (d) aerogel-Nomex nonwoven felt at $\times 30K$]

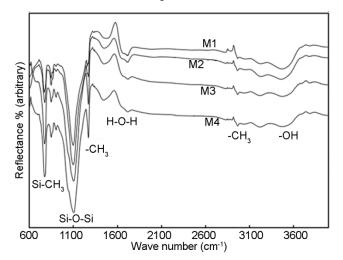


Fig. 2 — ATR-FTIR spectra of MTMS based aerogel-Nomex nonwoven composite felts

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reactions, leading to a superhydrophobic/flexible aerogels. The resultant aerogel-Nomex nonwoven felts using MTMS precursor in this experimental work has been found to retain a certain degree of flexibility, which can be observed from Fig. 3, where normal (without aerogel) and aerogel-Nomex nonwoven composite felts are allowed to bend by applying a small load at one end.

3.1 Bulk Density and Thermal Conductivity

The bulk densities of all the three-layered fabrics and aerogel-Nomex nonwoven composite felts are determined by taking the weight of samples on an electronic microbalance, and from the measurement of dimensions (length, width and thickness) of the aerogel-Nomex nonwoven felts samples, using the formula given earlier (Eq. 1). The thicknesses of all the aerogel-Nomex felts have been determined on an electronic (thickness) tester at a pressure of 2000Pa. The values of experimentally measured thickness, calculated density and thermal resistance of the aerogel-Nomex nonwoven fabrics are given in Table 1. The data (Table 1) show the effect of changing the molar ratio M (Methanol : MTMS) on bulk density and thermal resistance for the composite aerogel felts.

The parent Nomex nonwoven fabric has bulk density of 52.0 kg/m³, whereas the bulk density of the lightest aerogel-Nomex fabric is found 78.8 kg/m³, which is much greater than the parent nonwoven fabric. It is observed that with increasing the methanol proportion in the sol for the samples from M4 to M1, it causes less amount of gel to be formed inside the fabric, and the gelation time is also much greater for samples M1 and M2. The dilution of the sols (higher MeOH: MTMS molar ratio), is the probable reason for lower bulk densities of the composites. In spite of increased bulk density, the measured thermal resistance of the aerogel-fabric samples has been found to increase with increase in precursor

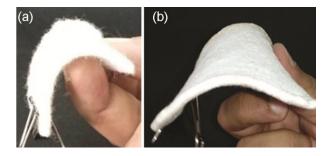


Fig. 3 — Flexibility in nonwoven fabric before and after formation of aerogel (a) Nomex nonwoven fabric and (b) aerogel –Nomex nonwoven composite

proportion, which can be due to greater thickness, blockage of thermal radiation and reduced gas phase conductivity owing to the presence of the aerogel.

3.2 Radiant Heat Protective Evaluation

The radiant heat protective performance of three layer clothing assemblies [OL-(M1-M4)-IL] comprising Nomex-III woven cloth as an outer layer (OL), an aerogel-Nomex nonwoven composite flexible insulation felt as a middle layer (M1-M4) and modacrylic-cotton (60:40) woven as inner layer (IL) has been estimated by determining the protection time exposing to a radiant heat intensity (35.0 kW/m^2) , based on Stoll curve. As the human skin is exposed to a given heat flux a certain amount of heat must be absorbed, causing damage of the dermis and epidermis, and a blister eventually appears. The Stoll's curve has been constructed based on the experimental work of Stoll¹⁷, which relates the amount of thermal energy that must be absorbed in a given time to cause a second degree burn injury. As the calculated cumulative thermal energy obtained using Eq. (3), meets or just exceeds Stoll's curve on continuous heating, a second degree burn injury is predicted and noted as the protection time. The estimated or predicted protection time is obtained for all the fabric combinations and plotted against changing molar ratios (M) (Fig 4). It has been observed that for any combination, aerogel-fabric

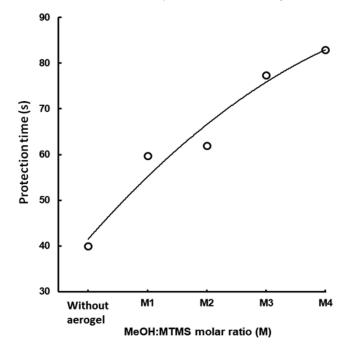


Fig. 4 — Changes in protection time (s) for different fabric combination with decreasing molar ratio of MeOH:MTMS in aerogel at middle layer

provides superior thermal insulation, in terms of protection time in seconds compared to the parent fabric combination; protection time increases rapidly with increasing concentration of MTMS. A maximum increase in protection time, as compared to the control fabric combination, has been found to be 107.3%, where aerogel-Nomex nonwoven felt is used in the middle layer.

In general, the higher thermal protection time, observed using the aerogel-Nomex nonwoven fabric combinations, is primarily due to the effect of aerogel formed inside the nonwoven fabric. The heat transmission through the aerogel particles surrounding the fibrous network is deferred by many mechanisms. It is known that optically thin aerogels are nearly transparent for the wavelengths smaller than 8µm, and hence it is likely that it behaves as a semitransparent material²¹. The aerogel-Nomex nonwoven felt, which is a composite material of silica aerogel within the fibrous matrix, can be described as an optically thick layer of material. Moreover, the Nomex III (m-aramid material) woven fabric used as the outer layer, have negligible radiative transmittance beyond the wavelength 2.5 µm (ref. 22). The effective or the total transmittance of thermal radiation for such an aromatic polyamide fabric determined on a spectrophotometer is found to be approximately 15% or $less^{23}$. Therefore, the heat transfer process through the aerogel nonwoven fabric can be considered as conduction dominated. The solid phase heat conductivity through the aerogel particles is dictated by the density dependent and geometrical factor that include ineffective dead ends of the solid chain like structural elements²¹. The characteristic pore size within the aerogel is well below the micron range, which causes gas phase heat transfer to be reduced, as compared to the free air. In conjunction with the reduced solid thermal conductivity and suppressed gas phase heat transfer, the aerogel-Nomex nonwoven composite felt acts as an excellent thermal insulating material.

The sample with highest proportion of MTMS (M4, molar ratio 15.4) results in highest protection. Among all the aerogel based-Nomex nonwoven fabric samples, highest thickness, more thermal resistance and bulk density are observed for M4 type sample. Hence, higher protection time is expected to be achieved, due to middle layer (Table 1).

The overall higher heat protection obtained using MTMS based aerogel felt can be attributed to greater thickness, increased opacity, porous nonwoven fabric

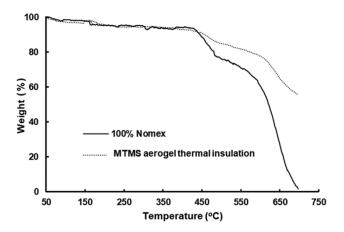


Fig 5 — TGA of a aerogel-Nomex Nonwoven insulations compared with 100% Nomex nonwoven felt

being blocked by aerogel, higher thermal resistance and superior thermally stability of aerogel nonwoven insulation (Fig. 5). The TGA study suggests that at about 700°C, 55% of the original mass of the composite is retained, whereas control Nomex fabric has got completely destroyed.

4 Conclusion

The flexible aerogel-Nomex nonwoven composite felts have been produced, characterized and evaluated for heat protective performance, using such a composite fabrics as thermally insulating middle layer in combination with other heat protective clothing layers. Superior radiant heat protection of 3-layer fabric has been achieved when MTMS based aerogel-Nomex nonwoven composite is used in the middle layer as compared to the usual combination of protective clothing layers. Better protection has been found with increasing MTMS concentration. Greater thickness, bulk density, higher thermal resistance, opacity, and thermal stability can be considered as the reasons of better radiant heat protection of such aerogel-Nomex composite materials. The flexibility achieved in composite insulation layers, is an added advantage to higher protection with MTMS type precursors. The Nomex-aerogel based fabric assemblies produced in these studies have the problem of dust shedding, which can probably be overcome using multilayered clothing assemblies and by application of suitable coatings.

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