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PREPARATION OF FLAME RESISTANT RUBBERISED COIR

By

A. COOMARASAMY

INTRODUCTION

The use of coconut fibre (coir) as a filling material for upholstery has been known for a long time. It is only during the 1950's that the possibility of utilising rubber latex in combination with coconut fibre or animal hair for producing cushioning materials with properties such as high resilience and elasticity was first recognised in Europe (Sirinivasan, 1964, and Kejariwal, 1977) this industry expanded rapidly especially upto the 70's (until the widespread use of polyurethane foam). The rubberised coir is mainly used as cushioning pads in mattresses, car seats and upholstery, as a packaging material and in insulation (Sirinivasan, 1964, Kejariwal, 1977). It has been stated that although several kinds of fibres such as Algerian grass fibre, sisal, Mexican fibre can be used, coconut fibre has proved by far, the best material for the manufacture of rubberised fibre due to its high strength (Kejariwal, 1977). Though Sri Lanka is one of the leading producers of natural rubber latex and coir, it was only about two decades back that the commercial manufacture of rubberised coir commenced in Sri Lanka. These products are mainly manufactured, at present, by a few large companies/manufacturers using semi automatic production lines and by small industrialists at a cottage industry level. There has been a rapid growth in production of these products during the last decade in Sri Lanka.

But one of the main concerns of manufacturers as well as the consumers is the possible fire hazard of these products. Both the coir and the rubber are flamable materials and the open structure of the rubberised coir facilitates rapid combustion (Holker, 1980).

Due to the fire hazard of the rubberised coir and the new regulations regarding the safety in motor vehicles, such as the American Motor Vehicle Safety ((Holker, 1980) Standard 302 test for flammability (MVSS 302) of 1971 (Motor Vehicle Safety Standard) the use of the coir in vehicle cushioning has been discontinued in many countries. Hence it has long been felt necessary to develop suitable treatments for improving the fire resistance of these products. Some studies have been carried out by workers in other countries especially in the U.K. In one of these studies natural rubber latex prepared by treating with bromotrichloromethane, in the presence of radical initiators has been shown to be a suitable flame retardant binder for the manufacture of these products (Pendle ; Pole). This approach though very effective is not economical (Holker, 1980). In other studies, use has been made mainly of proprietary flame retardants for the treatment of coir, followed by the use of unmodified latex containing suitable additives. Work has also been done on the use of treated coir with blends of NR and polychoropene latex (Holker, 1980; Bone and Edwards, 1980).

However a considerable amount of work has already been done in the past by scientists and technologists on flame retardants for elastomers. The following types of compounds have been used most often in elastomers (Laewson, 1986). Chlorinated paraffines, polychlorinated alicyclics, brominated aromatics (particular by others), organo-halo phosphates and organophosphates and phosphites are the main organic types. Aluminium hydroxide, magnesium carbonate, antimony trioxide, iron oxide, zinc oxide, clay, silica, calcium carbonate, zinc borate, ammonian phosphate halides are the main inorganic types. As a lot of information is already available in the literature on flame retardant additives it was felt that it should be possible to carry out work on formulations to arrive at cost effective simple treatments to promote the manufacture of flame resistant rubberised products in Sri Lanka. The present paper is concerned with the work on the use of simple compounds as flame retardant additives in the manufacture of rubberised coir products.

Fire behaviour polymers

The combustion of a polymer involves a series of stages :—

In the first stage heat from an external source is transferred to the polymeric material causing its temperature to rise. At high temperature polymeric materials undergo thermal degration, resulting in the formation of pyrolysis gases and char (De S. K.)

Pyrolysis of most polymers involves the formation of two zones; pyrolysis zone, which moves into the solid away from the surface, as the material is heated and decomposed into gases and char ; and a porous char zone between the exposed surface and pyrolysis zone. Thermal degration occurs in the pyrolysis zone, producing gases which flow through the porous char into the surrounding gas. If the diffusion rate of oxygen (or air) is sufficient, it may enter into the char zone and react with the pyrolysis gases within that zone. Otherwise the final products of pyrolysis, mix with oxygen in the outside gas phase region to produce a flammable gas mixture. In that case the burning can be described as continuous diffusive gas phase ignition. The nature of the decomposition of products and their relative proportions are mainly dependent on the chemical nature of the polymeric material.

The fire behaviour of these two polymeric materials, natural rubber and coir are quite different. Coir which is a lingo-cellulosic material, on pyrolysis produces initially a tar rich leavoglucosan and less amounts of volatile compounds (Holker, 1980). These substances on combustion ultimately yield carbonmonoxide, carbondioxide and water with a small amount of carbaronaceous matter (char). Natural rubber on the other hand on pyrolysis, yields mainly volatile hydrocarbons and sulphur compounds mainly derived from the vulcanising agents, leaving very little residual materials. The volatile hydrocarbon burn readily to produce carbonmonoxide, carbondioxide, and water.

Flame retardants

It is evident from the above description that there are essentially two regions in the burning zone of the polymeric material during combustion ; The gaseous phase-flame and the condensed phase-char. The mechanism of fire retardancy therefore, could involve the suppression of conditions favouring combustion in both regions. Although the use of certain additives as flame retardants has been developed from empirical methods in the earlier days, at present the mechanisms of action of these additives and flame retardants have been well established (Fardell and Lucas, 1987).

Flame inhibitors operate by either physical or chemical mechanism or in a few cases both. Chemical inhibitors are much practical but materials such as water, inert gases should be mentioned as effective coolants/diluents. Chemical flame retardants alter the rate of reactions by interfering with a crucial step in the burning process. Halogen compounds and substances such as sodium bicarbonate, metal oxides such as antimony trioxide in the presence of halogen compounds operate mainly by this mechanism.

Condensed phase inhibitors are believed to act by retarding the fuel to the flamet. This is accomplished by either a reduction on the heat of pyrolysis or by catalying of cross linking reactions (at the expense of chain cleavage reactions) to enhance residue or char formation. Here again physical inhibition is possible by the addition of inert materials. Their mode of inhibiting combustion is exactly analogous to that of inert gases in the flame.

Char formation is significant on sections or materials of reasonable thickness. Char also may be induced by the addition of intumescent materials such as borates and phosphates. It has been stated that the char promoters which are quite effective with cellulosic materials, do not work well with polymeric hydrocarbons. The conventional approach with these materials is to add halogenated flame retardants which act in the vapour phase and when these are used as the sole retardants the level required to give self extinguishing characteristics is about 15% bromonine or 40% chlorine. The addition of a synergist such as antomony trioxide allows more reductions in the quantity of flame retardants. However, in many cases such as the American Motor Vehicles Standard 302 test for flammability (MVSS 302 test) it is sufficient to achieve a reduction in burning rate rather that the material should be self extinguishing, in which case a reduction in the quantity of retardants if possible (Bone and Edwards, 1980).

EXPERIMENTAL AND REULTS

The compounded latex required for the preparation of rubberised coir samples was prepared according to the following formulation.

Table 1. *Vulcanisation formulation*

Ingredients	Wet weight g	Dry weight g
60% High Ammonia latex	167·00	100·00
10% Vulcastab L.W. solution	50·0	0·50
20% KOH solution	2·5	0·50
50% ZnO dispersion	10·0	5·0
50% ZDC dispersion	2·0	1·0
50% ZMBT dispersion	1·0	0·5
50% S dispersion	6·0	3·0
50% Antioxident dispersion	2·0	1·0
Clay slurry (in the absence of flame retardants)	20·0	10·0
Flame retardant	variable	variable
Water (to adjust viscosity)	variable	variable

Preparation of rubberised coir pads for testing

The coir fibre required for the experiments was obtained by loosening the curled fibres manually from a twisted rope. The loose fibre (45 — 50g) was spread out and packed uniformally by hand, in a rectangular wooden frame of dimension 19·5″×3·5″X 1″. Compounded latex (100ml) was then sprayed uniformally on both sides of the pad with the help of a spray gun. The pad was then dried and vulcanised for a period of 15 mts at 70°C in an air circulating oven. The pad was then removed from the wooden frame and post vulcanised at 50°C for 2 hours. Subsequently the pads were stored in a humidity controlled (RH 50 — 55 %) room.

Treatments used

The pads were prepared using untreated curled fibres with compounded latices containing chemicals such as borax, chlorinated hydrocarbon (Cereclor S 45, ICI Ltd.) antimony trioxide, calcium pyrophosphate, fluro-apatite, hydrated aluminium oxide, polyvinyl alcohol and polyvinyl alcohol-boric acid adduct.

Rubberised coir pads were also prepared using curled fibre which has been treated with either borax or boric acid solution or a thin dispension of calcium pyprophosphate. The compounded latices used in these experiments too contained the flame retardant additives mentioned above. In one instance, natural rubber latex which has been chemically modified with bromotrichlomethane at 15 phr (in our laboratory) was used as the binder after suitably compounding it.

Evaluation of flammability

1. Qualitative evaluation

Two inch strips of rubberised coir from various samples were tested (individually) by holding a bunsen flame at one end while keeping them horizontically and the qualitative observations as regards their flammability were made by checking whether the samples burnt fully. Samples which showed resistance in this evaluation were then tested according to the MVSS 302.

2. Quantitative flammability testing

Flammability tests were carried out in accordance with the procedure set out in Motor Vehicle Safety Standard No. 302 (USA Department of transportation 1971) except that instead of the special cabinet, a fume cupboard chamber was used.

The pad cut from a specimen conditioned at 50 —55 % relative humidity (RH) was mounted horizontically on a frame inside the fume cupboard chamber, a bunsen flame 1·5 inches in height with air inlet to the burner closed, was applied to the open end of the specimen for exactly 15 seconds and extinguished. The time taken for the flame to travel between two marks 10″ apart was noted and the burn rate was calculated as follows :

$$\text{Burn rate} = 60 \times \frac{\text{Distance travelled (inches)/min}}{\text{Time taken (in seconds)}}$$

The test specifies a maximum burn rate of 4" per minute. If a material stopped burning within 60 seconds from the start of timing and had not burnt more than two inches the material was considered to meet the requirements of the test.

The results on the qualitative evaluation of pads prepared with untreated coir and compounded latex containing various additives are given in the Table 3. Thse reults obtained on qualitative tests with pads prepared with treated coir fibre and compounded latex containing fire retardant chemicals are given in the Table 2.

Table 2. *Qualitative assessment of flammability of rubberised coir prepared with untreated curled coir fibre and compound latex containing flame retardant additives*

Additives in latex (chemical)	Quantity phr	Flammability qualitative
None	–	burnt rapidly
Borax	5	burnt rapidly
Cereclor S 45	6	burnt rapidly
Antimony trioxide	20	self extinguishing
Antimony trioxide	10	self extinguishing
Calcium pyrophosphate	10	burnt slowly
Calcium pyrophosphate	20	burnt slowly
Calcium pyrophosphate	30	partially burnt
PVA/boric acid adduct	10	self extinguishing
PVA/boric acid adduct	3	burnt slowly
Hydrated aluminium oxide	15	burnt leaving a char
Calcium carbonate	20	burnt with a smoke
Flouro apatite	15	burnt rapidly

Table 3. *Qualitative assessment for flammability of rubberised coir prepared with treated fibre and rubber latex containing flame retardant additives*

Chemical used for treatment of coir	Additive to latex (chemical used)	Quantity phr	Flammability qualitative
Boric acid	—	—	burnt fast
Borax	—	—	burnt rapidly
Borax	Borax	5	burnt rapidly
Borax	Cereclor	6	burnt rapidly
Borax	Antimony trioxide	30	slightly charred on the surface
Borax	Antimony trioxide	20	slightly charred on the seurfac
Borax	Antimony trioxide	10	self extinguishing
Borax	PVA	10	burnt slowly
Borax	Calcium pyrophosphate	10	self extinguishing
Borax	$CBrCl_3$ modified latex	15	self extinguishing
Calcium pyrophospate	PVA/Boric adduct %		burnt slowly leaving a char

From the results it can be seen that the pads made with untreated fibre and latex containing compounds such as antimony trioxide, calcium pyrophosphate and PVA-boric acid adduct have certain amount of resistance towards rapid combustion. The results obtained (Table 2) also clearly show that treatment of fibre (coir) infact improves the resistance towards flammability.

The results on the quantative tests carried out according to the MVSS 302 on selected samples are given in the Table 4.

Table 4. *Formulations satisfying MVSS 302 test for flammability*

Chemicals used for treatment of coir	Additive to latex (chemical used)	Quantity phr	Time for self extinguishing (seconds)	length of spread (inches)
Borax	Antimony trioxide	10	10 seconds	less than 1/2″ on the sample
Borax	Antimony trioxide	30	a few seconds	slightly charred only on the surface
Borax	Calcium pyrophosphate	10	20 seconds	less than 1/2″ on the sample
None	Antimony trioxide	30	a few seconds	slightly charred on the surface
None	Antimony trioxide	20	a few seconds	slightly charred on the surface
Borax	$CBrCl_3$ (for modification of latex)	15	30 seconds	1/2″ on the sample
None	PVA/Boric acid adduct	10	85 seconds	2″ on the sample

The results given in the Table 4 indicate that all the samples selected except the last one were self extinguishing within 60 seconds under the testing conditions.

From these results it can be concluded that flame resistant rubberised coir products which could satisfy MVSS 302 can be produced by the use of chemicals such as antimony trioxide and calcium pyrophosphate in latex. It has been found that the treatment for coir with borax enhances the flame resistant characteristics of the product ,which in turn makes it possible to reduce the quantity of antimony trioxide or calcium pyrophosphate added to the compounded latex used in the preparation of rubberised coir. These treatments are relatively inexpensive and therefore are attractive for these applications. PVA/boric acid adduct as an additive to latex too was quite satisfactory as regards imparting flame resistance but it was observed that the use of this additive resulted in a drop in physical properties of the material.

Our results on the use of bromotrichloromethane latex confirmed the findings of earlier workers that this treatment imparts flame resistance. However, the modified latex would be very expensive for these applications.

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