STANDARDISATION OF RADIATION VULCANIZED NATURAL RUBBER LATEX

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INTRODUCTION

Since the beginning of the Regional Cooperative Agreement (RCA) project on radiation processing in 1978 a wealth of information has been accumulated on radiation vulcanization of natural rubber latex. When it comes to technology transfer the process should be supported by quality assurance and it is necessary to formulate standards for testing. This paper attempts to describe standard techniques for the production of radiation vulcanised natural rubber latex (RVNRL).

STANDARD LATEX

The general purpose grades of natural latex concentrate are now subject to the requirements of international standards. They have been technically specified for many years in certain countries and the establishment of international standards indicates the substantial measure of agreement that has been achieved on the characteristics which are required of good quality natural rubber latex. International Standards Organisation (ISO) has defined the requirements for both centrifuged and creamed natural rubber latices[1].

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[1] A paper presented at the International Symposium on Radiation Vulcanized Natural Rubber Latex held at Takasaki, Japan in August 1989.

SAMPLING

The sampling method is described in an international standard[2]. Rubber latex may tend to cream on standing. If stractification has occured, the latex shall be thoroughly agitated to obtain a homogeneous blend suitable for the withdrawal of a representative sample. The procedure required differs with the type of container. In all cases where samples are drawn from several containers, 10 per cent sampling of latex in drums or in the case of tanks where extractions are made at various depths, such samples should be bulked and stirred. The final average sample shall then be bottled and sealed.

Standard specifications for centrifuged latex, both high ammonia (HA) type and low ammonia (LA) type are given in Table 1. Any additional characteristics of latex such as pH, specific gravity, viscosity, conductivity, particle size and particle size distribution would be useful. In the case of LA latex the type or stabilizer system should be specified.

STANDARD IRRADIATION CONDITIONS

Standard recommended practice for exposure of polymeric materials to high energy radiation is described in a standard[3] issued by the American Society for testing materials (STM). Irradiation conditions should be reported in the following manners :

1. Type of radiation source and kind of radiation
2. Irradiation dose rate, kilo gray per hour
3. Irradiation time
4. Total dose in kilo gray
5. Reference to or description of irradiation dose measurement procedure
6. Dimentional description of test specimen size/size of container. Specify the design/type of container used for irradiation and the design/type of stirrer needed.
7. Description of the type of material tested shall be reported including the nature of additives eg sensitizer, stablizer, emulsifier and the method of incorporation of the additives into the latex. The total solids level of the latex should be indicated.
8. Description of irradiation conditions such as the nature of the atmosphere in which irradiation is carried out, temperature of irradiation and the speed of stirring if stirring is carried.

It is necessary to add a sensitizer in a slow stream while the latex is stirred continuously. The sensitizer should get absorbed into the latex particles and at least 16 hours should elapse before the latex is irradiated. It is necessary to stir the latex at a speed ranging between 20 to 60 rpm during irradiation, especially for large batches.

FURTHER TREATMENT OF IRRADIATED LATEX

It is important to add an antioxidant to the irradiated latex at the rate of 1 to 2 parts per 100 parts of rubber. If the antioxidant is not soluble in water it should be added as a 50 to 80% dispersion in water. The recommended antioxidants are the non staining phenolic types given in Table11. However other antioxidants may be used depending on the type of application for which the irradiated latex is used.

STANDARD METHODS OF TESTING IRRADIATED LATEX

A few methods of testing irradiated latex are described.

1. Shelf life

   The viscosity of irradiated latex should be determined on a monthly basis. Depending on the increase of viscosity with time the storage stability can be graded as Excellent (E) Good (G) or Poor (P).

   The latex should be stored in well stoppered bottles under controlled temperature and humidity conditions. The viscosity should be measured under controlled conditions using a well known type of a viscometer. eg. Brookfield viscometer

2. Level of prevulcanization

   (a) In line quality control test:

   The chloroform coagulation test is a simple and rapid means for testing the degree of prevulcanization[4]. It consists of mixing equal parts of prevulcanized latex and chloroform (10 cm^3 of each) and stirring with a glass rod until coagulation is complete. The coagulum is allowed to stand 2 to 3 minutes and then numerically rated from 1 to 4. No 1 is judged as uncured and No 4 is precured to an advanced degree. The chloroform coagulation test is subjective and it does not allow for easy comparison of small differences between individual samples.

(b) Swelling test:

Solvent swell tests are more quantitative than the chloroform coagulation test.

A quick method has been developed to determine the degree of vulcanization[5].

A film of latex is formed on a specially treated paper (eg mita copy paper). The film 0.125 mm in thickness (after drying) is cut into a circle (a 50 mm diameter) with the paper tacking and placed in a medium size petri dish containing toluene. The film is allowed to swell for 20 minutes (until swelling is maximum) and the swollen diameter is measured. The percentage swelling gives an indication of the degree of vulcanization.

Unvulcanized	160%
Lightly vulcanized	100 – 160%
Moderately vulcanized	80 – 100%
Fully vulcanized	75%

3. Film formation

(a) Coagulant dipping

The following coagulant solution is recommended

Ingredients	Parts by weight
Calcium nitrate	25
Water	24
Methylated spirit	50
Wetting agent	1

A clean dry former is immersed in a coagulant solution and partially dried (at 70 °C) to leave a uniform layer of coagulant over its surface. It is then immersed in the latex at a steady rate allowed to dwell there for an appropriate time to obtain the required thickness. The wet gel deposit is leached in hot water (70°C – 80°C) for about 30 min. It is then dried at 70°C in an air circulating oven until moisture content is reduced to less than 0.5%.

This is followed by post – heat treatment at 100 °C for 30 min. It is then dusted. The film is then tested for physical properties.

(b) Straight dipping

In straight dipping a coagulant solution is not used. The rest of the procedure is the same as for coagulant dipping.

In studying the dipping characteristics the relation between the deposit thickness, dwell time and number of dips should be studied. The processing characteristics should also be noted from possible defects such as pin holes, webbing, lamintion etc. Other specifications required for specific end products such as the bursting pressure should be met.

TESTING OF FILM FOR PHYSICAL PROPERTIES

Preparation and pretreatment of film is necessary prior to testing.

1. Preparation of film for testing

Carefully strain the latex through a 180 m stainless steel sieve with a nominal apparature of 0.180 + 0.009 mm into a beaker.

Cover the beaker to minimise surface drying and allow it to stand for 5 minutes before pouring the latex into the glass mould.

The irradiated latex is cast on a glass mould (150 mm square or about similar dimension) and dried under normal atmospheric conditions. The thickness of the dried film should be about 0.75 mm. The film is leached for 24 hrs in water at room temperature and subsequently oven dried at 70°C for 4 hours. The film is then given post heat treatment at 100 °C for 30 min. The film is then dusted using talc.

2. Pretreatment of film prior to testing

(a) Leaching

The properties of dried films from prevulcanized latex are affected by leaching them in water and by heating. A pronounced effect on leaching is seen, giving rise to increased Tens ile Strength and Modulus.

The explanation of the leaching effect is thought to be in the ability of surface active agents and other hydrophilic materials in the compound to hinder the formation of a homogeneous film on drying. Since the molecular mobility is restricted by the crosslinks, particle merging becomes more difficult and is easily hindered by the non rubbers. Leaching

removes a large portion of these non rubbers and facilitate homogeneous film formation. Heating the film will assist the particles to merge but may introduce extra crosslinks or rearrange existing ones.

(b) Heat treatment

A significant improvement in the physical properties of the film derived from irradiated latex is evident after heat treatment of the prevulcanised film for 30 min at 100°C.

3. Testing of irradiated latex films

The testing procedures are described in the relevant ISO or British Standards (B. S.) and are given in Table[111]

REFERENCES

1. ISO 2004
2. ISO 123
3. ASTM–D–1672–66 (1971)
4. The Vanderbilt Latex Handbook, Third Edition, 110
5. P. F. Murray, NR Technology, 13, 31 (1983) Part 2
6. ISO 124
7. ISO 126
8. ISO 125
9. ISO 35
10. ISO 706
11. ISO/R 1654
12. ISO 1655
13. ISO 2005
14. ISO 506
15. ISO 127
16. ISO 471 – 1977 (E)
17. ISO 471 – 1977 (C)
18. ISO 37 – 1977 (E)
19. ISO 188 – 1982 (G)
20. ISO 2285 – 1981 (E)
21. ISO 34 – 1975 (E)
22. BS 1673 : Part 4, 1953

Table 1. *Standard specification of HA and LA Latex*

Characteristics	High Ammonia Latex	Low Ammonia Latex	Reference No.
Total solids content, 5m/m, min	61.5	61.5	6
Dry rubber content, 5 m/m, min	60.0	60.0	7
Non-rubber solids, % m/m, max	2.0	2.0	
Alkalinity (as NH3) % m/m, on latex concentrate	0.6 min	0.29 max	8
Mechanical stability, seconds, min	650	650	9
Coagulum content, % m/m, max	0.05	0.05	10
Copper content, mg/kg of total solids, max	8	8	11
Manganese content, mg/kg of total solids, max	8	8	12
Sludge content, % m/m, max	0.10	0.10	13
Volatile fatty acid No., max	0.20	0.20	14
KoH number, max	1.0	1.0	15
Colour on visual inspection	No pronounced blue or grey	No pronounced blue or grey	
Odour after neutralization with boric acid	No pronounced odour or putrefaction	No pronounced odour or putrefaction	

Table 11. *Some non staining antioxidant types with their trade names*

Type of Antioxidant	Trade example
Phenol alkanes and hindered phenols	Antioxidant 2246
	Antioxidant 425
	Nanga white
	Permanex SP
	Permanax WSL
	Permanax WSP
	Santowhite 54
	Vulkanox BKF
	Vulkanox ZKF
	Wingstay L
	Wingstay S
	Wingstay T
Hydroquinones	Agerite alba
	Santovar A.

Table 111 *Testing Procedures for Rubber Films*

Test method	Reference No.
Conditioning of test pieces	16
Methods of testing vulcanized rubber	17
Determination of tensile stress–strain properties	18
Accelerated ageing or heat resistant tests	19
Determination of tension set at normal and high temperature	20
Determination of tear strength	21
Procedure for testing swelling and gel fraction	22
Colour, transparency and clouding effects	–
Toxicity tests	–
Combustion analysis of films (gas evaluation and ash content)	–