

Cariflex[®]
Polyisoprene Products

Preparation of synthetic polyisoprene latex and its use in coagulant dipping



 **Kraton**
Giving Innovators Their Edge

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Rubber

Natural rubber is one of the best known natural polymers. The material is derived from a milky suspension (latex), found in the sap of some plants. Mayans and Aztecs already harvested it from the hevea tree, and used it to make waterproof boots and balls. It is an elastomer: it recovers its original shape after stretching or deformation.

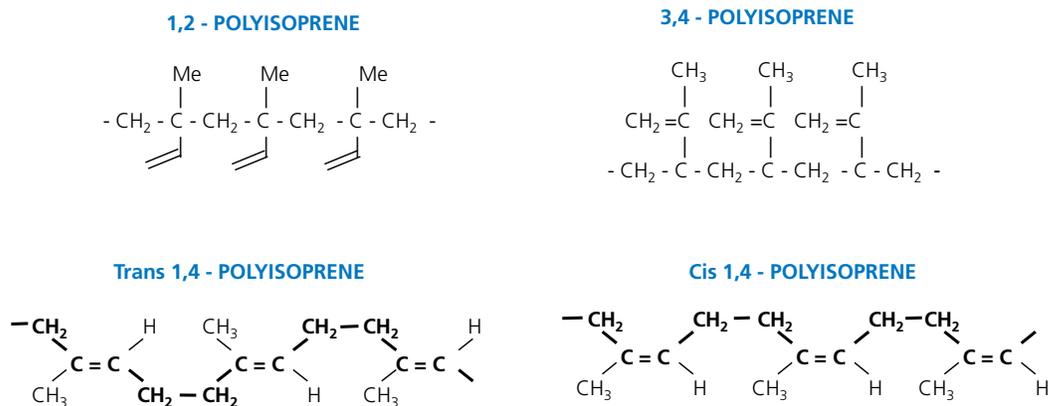
Natural rubber is a polymer of isoprene (2-methyl-1,3-butadiene) with a molecular weight of 100,000 to 1,000,000. Most often the isoprene consists almost completely of cis-1,4-IR (~99%). This high cis content gives rise to strain hardening. The long, entangled chains are considerably branched¹. Initially, natural rubber contains 5-10% of low gel. Upon storage the gel content increases and may reach values of 50%. Normally, natural rubber is treated to crosslink it.

Typically a few percent of other materials (proteins, fatty acids, resins and inorganic materials) are found in high quality natural rubber^{2, 3, 4, 5, 6, 7}. Investigations have shown that people may be sensitized to natural rubber latex allergens.

Polyisoprene

Polyisoprene elastomers (the synthetic version of natural rubber) are synthetic, predominantly stereoregular polymers that closely resemble natural rubber in molecular structure as well as in properties. Synthetic polyisoprene has a slightly lower percentage of cis-1,4 polyisoprene units. Manufacturing polyisoprene (IR) with the desired properties is not a trivial task: the polymerization of isoprene can lead to various repeating units. The four types that are possible (1,2-IR, 3,4 IR, trans-1,4 IR and cis-1,4 IR) are indicated in Figure 1.

Figure 1: The four types of polyisoprene. Cis-1,4 polyisoprene is the type present in natural rubber.



Several different processes can be used to prepare polyisoprene (see Table 1). Ziegler-Natta polymerization (coordination polymerization) allows cis-1,4 IR, trans-1,4 IR and 3,4-IR to be tailor made. This type of polymerization is the most widely used process to produce polyisoprene having a cis-1,4 content above 96%.

Table 1: Different processes to produce polyisoprene

Type of polymerization	Remarks
Coordination polymerization	Taylor made polymer; high cis, high trans or high 3,4
Anionic polymerization	High cis or medium 3,4
Radical polymerization	>65% 1,4 trans, low Mw
Radical emulsion polymerization	Low cis, high branching/gel, low conversion
Cationic polymerization	Built-in cycles, low Mw
Biosynthesis	in trees

Anionic polymerization results in a polymer with a cis-1,4 content of more than 90% as measured by NMR. This is the polymerization process that is employed by Kraton Polymers. This process can also be employed to produce 3,4-IR with a 3,4 content of about 60%.

Radical polymerization is not an option to make cis-1,4 polyisoprene so far. With this type of process mostly 1,4 repeating units are generated and only low molecular weight chains can be obtained at a reasonably high conversion rate. Radical emulsion polymerization is even more difficult to control. So far, this type of polymerization can unfortunately not be used to make a suitable polyisoprene latex in one step. Polyisoprene produced via cationic polymerization suffers from low molecular weight, and a high level of built-in cycles.

The process at the origin of natural rubber occurs in *Hevea Brasiliensis* trees. In this process the polyisoprene is produced as a latex (emulsion).

Relative polymer properties

Of the processes to make polyisoprene as given in Table 1, only three are possible to produce high cis-1,4 polyisoprene. Between the products produced by these three processes, there are clear and important differences (see Table 2). Natural rubber contains no synthetic catalyst. It is a branched polymer with a high molecular weight (MW) and a broad molecular weight distribution (MWD). The stereoregularity is extremely high with a cis-1,4 content of about 99%. Due to the combination of high MW and branching, natural rubber contains gel particles. The level of natural impurities, such as proteins, is in the order of 1% on solid rubber^{8,9}. Natural rubber is produced as an emulsion, and can be converted into a solid rubber.

Table 2: Different processes to produce polyisoprene

Natural Rubber	Zieger Natta IR	Anionic IR
No catalyst	Titanium/Aluminum catalyst	Alkyl-lithium catalyst
Branched product	Branched product	Linear products
Wide MWD*	Wide MWD*	High MW, narrow MWD*
98+% cis content	96+% cis content	90+% cis content
High gel content	High gel content	Intrinsically NO gel
Contains natural impurities	Catalyst residuals	Low impurity level
Produced as emulsion	Produced as organic solution	Produced as organic solution

* MWD: Molecular Weight Distribution, MW: Molecular weight

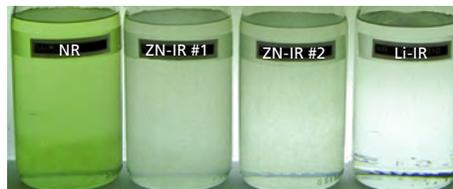
Ziegler-Natta high cis-1,4 IR (ZN-IR) is polymerized in the presence of a titanium/aluminum catalyst. The product is branched and has a wide MWD. Stereoregularity of this polymer is high. Although the molecular weight of this type of polymer is only about 1.000.000, the gel content is as much as 10 to 20%¹⁰. ZN-IR usually contains catalyst residuals.

The anionic polymerization of isoprene into anionic polyisoprene is initiated with an alkyl-lithium initiator. The resulting polyisoprene (Li-IR) is a fully linear polymer with a relatively narrow MWD. The molecular weight is about two to three times as high as that of ZN-IR. Li-IR has a gel level close to zero, and a very low level of metal residuals. Typically, the cis content of Li-NR is 91% (as measured by NMR).

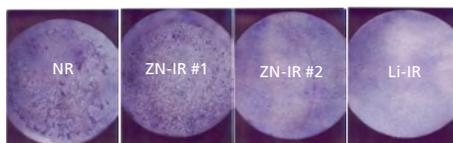
Both ZN-IR and Li-IR are produced in organic solutions, which can subsequently be coagulated and dried into solid rubber, or converted into latex via a post-emulsification step. The gel content may have an impact on the quality, and thus handling, of solutions, as can be seen in Figure 2, showing diluted solutions of natural rubber, ZN-IR and Li-IR. The ZN-IR #2 solution is based on a ZN-IR grade that has undergone an extra purification step. A settled gel layer can be seen in the natural rubber solution. Although it is not visible on this photograph, a similar layer is observed in reality in ZN-IR solutions. Wet-gel analysis gives evidence of the presence of gel in natural rubber and all ZN-IR samples. Standard Li-IR in solution is crystal clear and shows no evidence of gel in the wet gel analysis.

Figure 2: gel content of natural rubber, two different ZN-IR samples and Li-NR. The upper photographs show the gel content of the samples when dissolved in toluene. The lower photographs show the results of wet gel analysis.

4% solutions in toluene



Wet gel analysis



The differences between natural rubber, ZN-IR and Li-IR at the molecular level induce obvious macroscopic dissimilarities. These are shown in Table 3. Color and smell are better in Li-NR: natural rubber and ZN-IR have brown or yellow colors, and relatively strong smells, while Li-IR is clear and has a light smell.

Table 3: Post-emulsification process to prepare Cariflex® IR0401 latex from polyisoprene

	Natural Rubber (Ribbed Smoked Sheet #1)	Ziegler-Natta IR	Anionic IR
Mooney Viscosity	120	50 - 90	40 - 90
Limiting Visco Number	6.0 - 7.0	2.5 - 4.5	6.5 - 9.5
Color	dark/yellow-brown	yellow/amber	crystal clear
Smell	some	some	light/neutral
Green Strength	high	moderate	moderate/cold flow
Flow of compound	moderate	moderate	high
Shore Hardness/Modulus	high	high	low
Tensile Strength	high	moderate	lower

The green strength of Li-NR and ZN-IR are on a par¹¹, both significantly lower than that of NR. Because Li-IR is a linear polymer, it flows exceptionally well. The counterpart of this is cold flow during storage.

Since there is a direct link between cis-1,4 content and tensile strength, it is not surprising that vulcanized rubbers based on ZN-IR are slightly less strong than those based on NR, and slightly stronger than those based on Li-NR¹². On the other hand, Li-IR will have the lower moduli and the highest elongation on break. Especially the low modulus of Li-IR is a popular property because it results in increased softness of the material.

Table 4: Non polyisoprene content

Natural Rubber	Natural Rubber		ZN-IR	Li-IR
	NR Sample 1	NR Sample 2		
Ash [%wt]	0.50	0.15-0.30	0.15-0.30	0.05-0.10
Stabilizer [%wt]	2.0-3.0	0.5-1.0	0.5-1.0	0.05-0.3
Metal Content [ppm]				
Al	180	8	120-130	<1
Ti	4	<1	180-360	<1
Li	<1	<1	<1	2-5
Fe	116	12	3-10	2
Na	35	97	15-150	5
K	490	250	10	<10
Ca	625	10	10-160	<1
Mg	120	123	2-10	<1
Cu	2	<1	<1	<1
Zn	7	3	<1	<1
Cd	<0.5	<0.5	<0.5	<0.5
Pb	<5	<5	<5	<5
Total	>1579	>503	>340	>9

Purity is the most obvious advantage of Li-IR (see Table 4). Not only is the ash content low, but also the anti-oxidant level. This leaves freedom to the formulator. The total metal content is the lowest of all types of IR and NR. For polyisoprene, low catalyst residual is the best guarantee of stability, thereby explaining why the level of added anti-oxidant can be kept low in Li-IR.

The nice thing about Li-IR is that you can actually see the difference as indicated in Figure 3.

Figure 3: Li-IR (shown in the middle) is a clear product with a very low level of impurities. On the left is an example of natural rubber, on the right ZN IR is shown



Cariflex® isoprene rubbers (IR) are high molecular weight, anionically polymerized polyisoprene that can be processed in the same way as a natural rubber, including vulcanization. They combine the key qualities of natural rubber such as good mechanical properties with superior features such as high purity, excellent clarity, good flow and low gel content. Cariflex IR polymers are available as bales of rubber or as a latex.

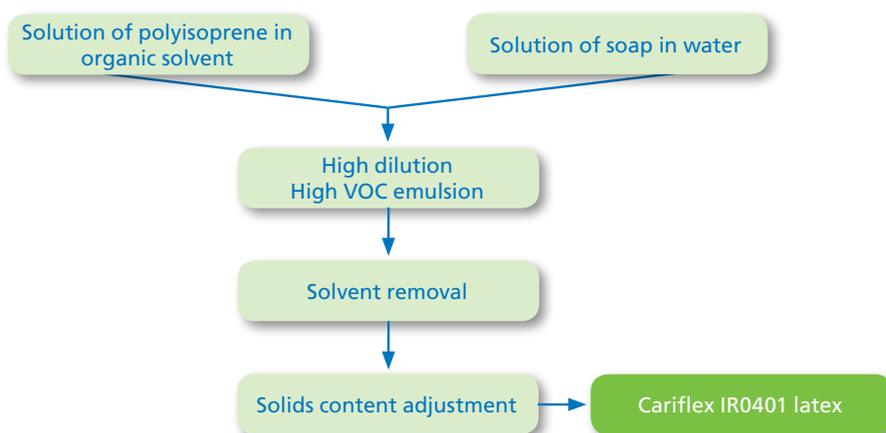
The very specific nature of Cariflex synthetic polyisoprene provides a number of factors that differentiate it from natural rubber. There is minimal variation in physical properties from batch to batch. Polymerization conditions are narrowly controlled to assure that the polymer is highly stereospecific in chemistry and has a narrow molecular weight distribution.

Polyisoprene latex

A polyisoprene latex can be prepared from the polymer via a post-emulsification process (see Figure 4). In this process, first the polymer is dissolved in a suitable organic solvent. This polymer solution is then blended with an aqueous soap solution in a high shear emulsification apparatus. The resulting intermediate product is a highly diluted emulsion with a high amount of organic solvent. The solvent contained in this emulsion is stripped, and subsequently the diluted emulsion is concentrated to produce the polyisoprene latexⁱ. The process parameters and know-how of the operators are key to the process.

The properties of our polyisoprene latex have been designed such as to match the ones of natural rubber latex (see Table 5). As can be seen the properties are much alike. One of the advantages of the artificial polyisoprene latex as compared to NR latex is that the impurity level of the former is lower (this is translated into a slightly lower rubber content of NR latex). In the artificial Cariflex IR0401(B) latex, the non-rubber content corresponds to the surfactant amount only. Furthermore, Cariflex IR0401(B) latex does not contain ammonia, and therefore no ammonia smell.

Figure 4: Post-emulsification process to prepare Cariflex IR0401 latex from polyisoprene



ⁱ. The product code of the Kraton Polymers synthetic polyisoprene latex is IR401 or IR401B.

Table 5: Typical Properties of Cariflex® IR0401

Latex	Cariflex IR0401 (B) Anionic IR	Natural Rubber
Total solids content [wt%]	65	Similar
Total rubber content [wt%]	64	Lower
pH	11	Similar
Ammonia [wt%]	0	0.2-0.8
Average particle size [μm]	1.5	0.9
Mechanical stability [sec]	>1500	>900
Brookfield viscosity [mPa.s]	50-150	Similar

Applications

Because of the good match of properties between the synthetic and natural rubber lattices, about the same wide range of technologies may be used, e.g. dipping, heat coagulation, extrusion of threads, etc. Cariflex IR0401(B) latex has a number of advantages as compared to natural rubber latex:

- It is a material with constant product quality;
- It does not contain ammonia;
- Cariflex IR0401(B) is produced from polyisoprene, a petrochemical source. As further, no proteins are used in its production process, we can expect within reason there are no natural rubber proteins present in Cariflex IR0401(B);
- Still, Cariflex IR0401(B) latex can be processed on existing natural rubber latex lines and it is compatible with all technologies (such as coating and sterilization) used with natural rubber latex. The strength achievable with Cariflex IR0401(B) latex is close to natural rubber and meets all official standards. Furthermore, IR401(B) has a low elastic modulus which gives these products a high comfortⁱⁱ.

One of the largest application areas for the use of Cariflex polyisoprene latex is in the area of thin-walled products, such as surgical gloves or condoms. The thin-walled products such as gloves or condoms are made through dipping, either straight- or coagulant dipping. In this brochure we will concentrate on coagulant dipping.

Products that can be obtained through the dipping process are not only thin-walled (0.02 to 2 mm), but have some typical properties that make the process favorable to produce gloves and condoms: elasticity and strength are usually both very high, and stretching to more than seven times the original length is possible.

Dipping

Dipped rubber goods are thin-walled products which are made by dipping a form in a latex. Latex then deposits on the form. The dipping is followed by drying and vulcanization of the film on the former. After the product is stripped from the form and washed, it is ready for use. The dipping process is a rather simple one. However, to be able to produce high quality products in enormous quantities, there are quite a number of important details to take care of. In the production of dipped goods a number of processes play equally important roles:

- The preparation of the compounded latex having the optimal composition;
- The actual process of dipping by which the form and thickness of the product is defined the vulcanization process that takes care of the required mechanical properties of the final product.

Apart from this, additional process steps may be needed, e.g. to modify the surface of the product.

Coagulant dipping: the process

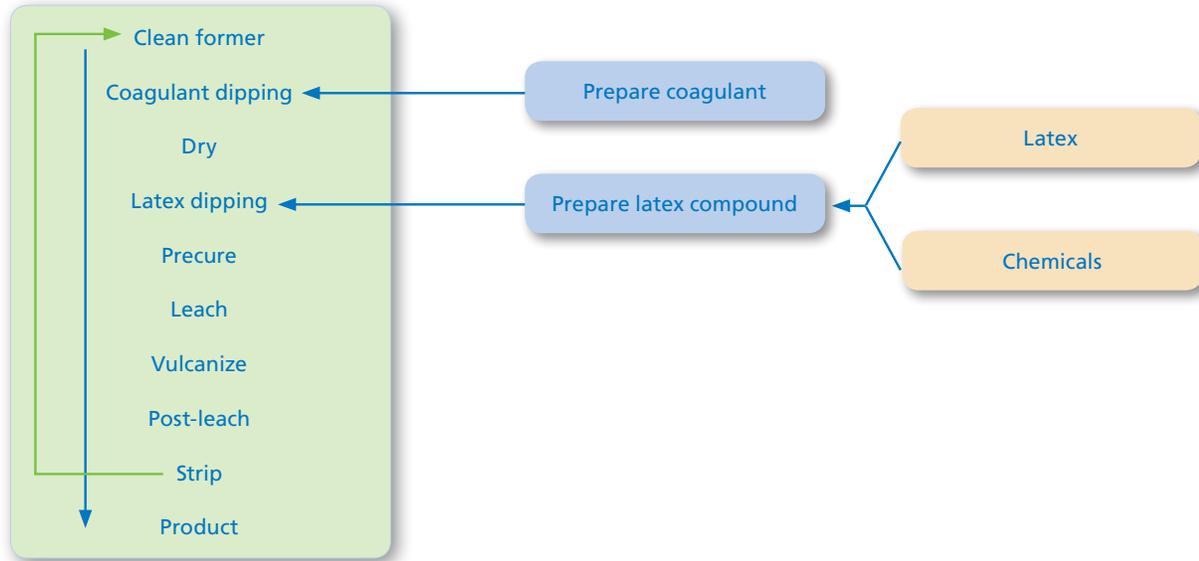
The different steps in the coagulant dipping process are depicted schematically in Figure 5. In this technique first a coagulant solution is prepared, examples are given in recipe 1. The latex is compounded with formulating ingredients, examples are given in recipe 2.

Prior to the actual dipping, formers are cleanedⁱⁱⁱ and then held in the coagulant solution. After drying of the coagulant on the former, it is held in the compounded latex for a certain dwell time. Now the compounded latex builds a layer on the former. After withdrawing it from the latex, the rubber coating is dried, leached, cured (vulcanized), and stripped off the former. The product may be completed if necessary, by subjecting it to appropriate post-treatments such as halogenation and/or sterilization. These post-treatments are not the subject of this brochure.

ⁱⁱ D.C. Blackley, "Polymer Latices, Science and Technology. Volume 3: Applications of Latices", 2nd edition, Chapman and Hall, London (1997), pp156

ⁱⁱⁱ Cleaning can e.g. be done by dipping the former into a weak acid solution and subsequently washing in hot water (Natuurrubber, 17, 1st quarter 2000)

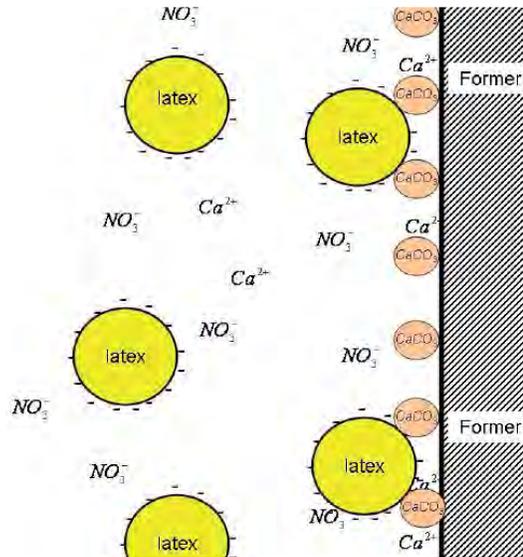
Figure 5: Schematic representation of the coagulant dipping process



Particle stabilization

Coagulant dipping depends critically on the type of stabilization of the latex particles. If the particles are stabilized ionically they can be destabilized by dissolved salts. In the coagulant dipping application, salts are deposited on a former by dipping the former in a salt containing coagulant solution, and subsequently drying it. When this former is then dipped into latex, the salts redissolve. Latex particles in the vicinity of the former experience a high salt concentration and therefore these particles are destabilized and form a layer on the former. The process is shown schematically in Figure 6.

Figure 6: cartoon of the coagulant dipping mechanism. In this example the latex particles are stabilized by negative charges. The particles are destabilized by nitrate and calcium ions. The insoluble calcium carbonate deposited on the surface of the former acts as a release agent when the dipped good is stripped off the former.



It is clear that the stabilizer of the (compounded) latex plays a large role in coagulant dipping. Cariflex® IR0401(B) latex polymer is stabilized with an anionic surfactant. It is the key to the stability to ensure that the pH of the raw material and of the compound remain above 10. Cariflex IR0401(B) latex polymer is not stabilized by the same surface active agents as natural rubber. Addition of extra stabilizer to Cariflex IR0401(B) latex may be required before compounding. A stabilizer often used in coagulant dipping applications is caseinate ((Na, K, or NH_4), which has the advantage of being very robust in the dipping process. For applications in which ingredients of animal origin should be avoided, the use of anionic surfactants (e.g. sulfates or sulfonates), or nonionic or amphoteric surfactants, offers an alternative. It should be kept in mind that the type of stabilizer used, may have an influence on behavior of the latex in the application. For example, when using a nonionic stabilizer, this may prevent the latex from forming a decent deposit in a coagulant dipping application, as particles stabilized by nonionic surfactants cannot be destabilized by high salt concentrations.

Ingredients that can be used in conjunction with natural rubber latex may sometimes destabilize Cariflex® IR0401(B) latex polymer, and vice versa. In case of stability problems, it is recommended to carefully screen the ingredients.

Leaching of the dipped goods

The leaching step ensures the removal of water-soluble material from the film (e.g. salts or surface active material).

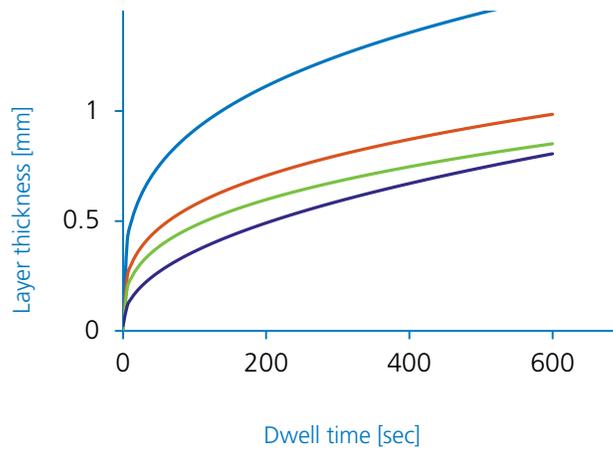
Final thickness of dipped goods

One of the important characteristics of the dipped good is its final thickness. The thickness of the deposited layer can be increased by increasing the dwell time in the compounded latex, increase the concentration of compounded latex, or by repeating the dipping process. The concentration of the coagulant solution and the dwell time in it only play a minor role in the final thickness of the deposited layer.

Typical examples of thicknesses of dipped goods as a function of dwell time in the compounded latex are shown in Figure 7 for different latex concentrations. For all concentrations of the compounded latex, the thickness of the dipped good can be described by a square root function of the dwell time. This is in accordance with what has been described in literature¹³ and is probably explained by the diffusion of the coagulant salt ions from the former to the water phase. At equal dwell time, the thickness increases as the concentration of the latex is increased. By selecting an appropriate latex concentration and dwell time a large variability of thicknesses can be achieved.

Figure 7: Thickness of dipped goods as a function of the dwell time in the compounded latex and of the concentration of latex.

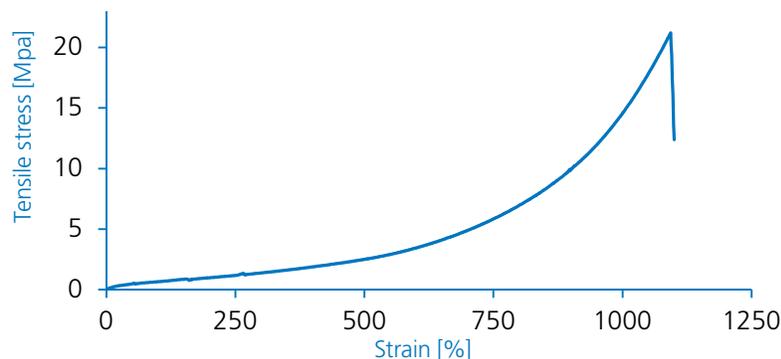
Latex concentrations were —: 60%, —: 50%, —: 40%, —: 30% weight%. The latex was stabilized with 0.8 phr sodium caseinate.



Mechanical properties of dipped goods

A typical stress-strain curve of vulcanized polyisoprene rubber is shown in Figure 8. As stated before, one of the unique properties of polyisoprene is its mechanical properties: high tensile strength, high elongation at break, and low modulus at elongation up to 700%. The latter is responsible for its softness and helps to provide a high degree of comfort.

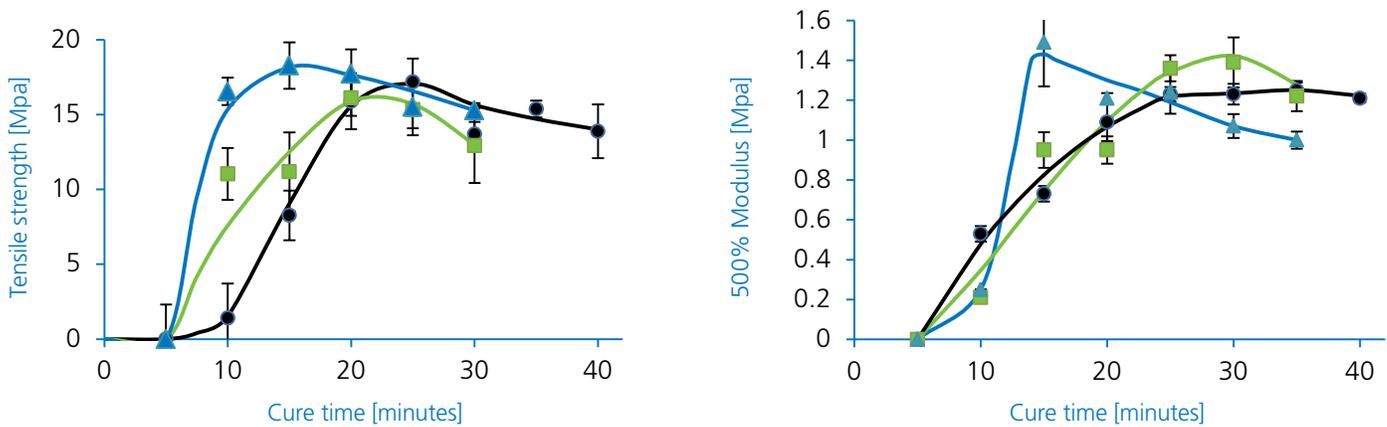
Figure 8: A typical stress/strain curve of a cured Cariflex IR0401(B) latex sample. The flat curve up to a strain of about 700% means that the material is soft.



Mechanical properties of the final dipped goods depend, amongst others, on maturation time of the compounded latex, and time and temperature of the curing step.

In Figure 9 both the tensile strength and 500% modulus are shown as a function of cure time for different cure temperatures.

Figure 9: Tensile strength and 500% modulus as a function of cure time for different cure temperatures. Experiments performed on compounded latex that was matured for one day. ●: cure at 110 °C, ■: cure at 120 °C, ▲: cure at 130 °C



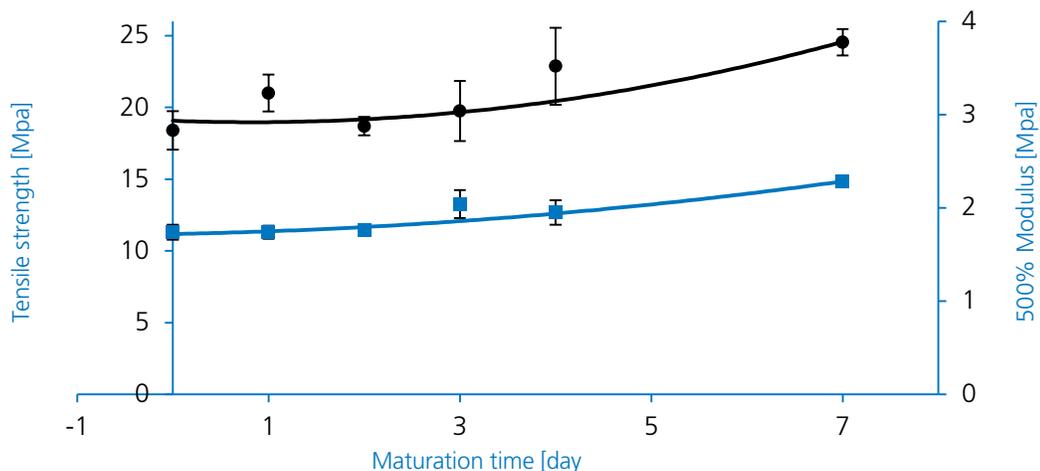
For all three cure temperatures tested, the tensile strength increases to a maximum and then decreases again. It is clear that the higher the cure temperature, the faster the maximum is reached. At a cure temperature of 130 °C, the maximum tensile strength can be achieved within a curing time frame from 10 to 20 minutes. This allows for a robust procedure.

The maximum of the 500% modulus is reached between 15 and 20 minutes cure time at 130 °C. At longer times the modulus decreases again. For the lower cure temperatures, the modulus reaches its maximum at a longer cure time, and it doesn't seem to decrease within the experimental window tested. In applications with different vulcanizing ingredients or concentrations, other cure times or temperatures may be necessary.

It should be taken in mind that apart from the curing at elevated temperatures, the process also takes place at room temperature, although this is much slower. Thus, e.g. a sample cured for 5 minutes at 130 °C, which is initially without tensile strength, may achieve mechanical properties comparable to a sample cured at 130 °C for 15 minutes, when stored at ambient temperature for several days.

In Figure 10 both the tensile strength and 500% modulus are shown as a function of maturation time. The dipped samples were cured during 15 minutes at 130 °C. As can be seen in Figure 10, the mechanical properties do not vary much over a couple of days of maturation, and already after a couple of hours good properties are achieved. Tensile strength and 500% modulus both increase with time. It was observed that when higher concentrations of vulcanizing chemicals were used, tensile stress reached a maximum after a couple of days and then decreased. This was explained by the following mechanism: during maturation, crosslinking already starts within the polyisoprene latex particles. During dipping and subsequent vulcanization, particles flow into each other and a coherent film is formed. The particles lose their identity. When crosslinking within the particles goes too far, film formation will be imperfect, and crosslinking will only occur to 'glue' individual particles together, but particles will not merge.

Figure 10: Mechanical properties as a function of maturation time. ●: tensile strength; ■: 500% modulus. Samples were cured at 130°C for 15 minutes.



As stated before, the selection of a stabilizer is very important. However, the type of stabilizer may have an influence on the properties that can be achieved with a certain formulation. We found that in the formulation as given in appendix (recipe 2), sodium caseinate could be used without affecting the mechanical properties of the dipped goods. However, in some cases it may be disadvantageous to use caseinate because it is of animal origin. As an alternative stabilizer, sodium dodecyl sulfate (SDS) was investigated. It was found that addition of 0.4 phr of SDS to the compounding formulation in appendix (recipe 2) had a negative influence on mechanical properties. Tensile strength reached a value in the order of 12 MPa after one day of maturation, as compared to a typical value of 20 MPa for a caseinate stabilized formulation.

The 500% modulus was also lower for the SDS containing formulation as for the one without. This loss of mechanical properties could be compensated by increasing the amount of vulcanizing chemicals. When these were doubled as compared to appendix (recipe 2), tensile strengths were again of the order of 20 MPa after one day of maturation.

Conclusion

Cariflex® IR0401(B) is an artificial latex that has a constant product quality and can be processed on the same type of equipment currently used for natural rubber processing with no required modification. Cariflex IR0401(B) is produced from polyisoprene, a petrochemical source. No proteins are used in its production process, so we can expect within reason there are no natural rubber proteins present in Cariflex IR0401(B).

Cariflex IR0401(B) latex can be used in coagulant dipping applications to prepare thin-walled products, such as surgical gloves. It is easy to compound Cariflex IR0401(B) latex with the compounding ingredients usually employed in vulcanization. Properties of the dipped goods depend on several process parameters, such as the dwell time in the coagulant solution and in the compounded latex, and on the temperature and residence time in the curing oven(s).

Not only is it possible to meet all specified requirements for surgical gloves with Cariflex IR0401(B), dipped products also can show a low elastic modulus which gives these products a high comfort.

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Appendix

Recipe 1: preparation of coagulant solutions

The function of the coagulant is to destabilize the latex particles: the particles deposit onto the former and form a film. The main components of the coagulant solution are the destabilizing salt (e.g. $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ or CaCl_2), and optionally a component that eases the release of the finalized product from the former. CaCO_3 is a well-known component that fulfills this job. The coagulant can either be water-based or contain an organic, volatile solvent. For a purely waterbased coagulant the drying is usually done at elevated temperature. An organic solvent based coagulant evaporates quickly at ambient temperature, but the organic solvent is released into the atmosphere. An example of a water-based coagulant is a solution/dispersion of 13.5 weight% $\text{Ca}(\text{NO}_3)_2$ and 5.5% weight% of CaCO_3 in water.

A solvent-based coagulant can be prepared by dissolving/dispersing 30 weight% $\text{Ca}(\text{NO}_3)_2$ in a mixture of 55 weight% iso-propanol and 15% water. For both types of coagulant solutions a wetting agent (e.g. 0.5 weight% Dresinate 214) may be beneficial. Typically the former is dipped for 20 to 40 seconds in the coagulant solution and subsequently dried.

Recipe 2: example of a compounded latex

As a general rule, the same additives as those used for natural rubber latex compounding can be utilised to compound Cariflex® IR0401(B) latex polymer. However, since it somewhat differs from natural rubber latex, such additives often must be properly selected, and added with care in order to benefit fully from the excellent properties of Cariflex IR0401(B) latex polymer. To make sure that compounding ingredients are mixed well with the latex, and to avoid subsequent settling out, water-soluble ingredients are added to latex as aqueous solutions and water-insoluble ingredients are added as aqueous dispersions or emulsions. De-ionised or soft water should be used. A vulcanization package usually contains the following ingredients:

- Accelerator: With Cariflex IR0401(B) latex polymer, the same vulcanisation accelerators as for natural rubber may be used but in somewhat higher quantities.
- Activator: a small amount of ZnO will suitably vulcanise Cariflex IR0401(B) latex polymer.
- Sulphur: compared with natural rubber latex, similar or smaller quantities of sulphur may be added to Cariflex IR0401(B) latex polymer. However, to maintain the same degree of tear and ageing resistance as those of other latices, it is recommended to use smaller quantities of sulphur.
- Anti-oxidant: the same anti-oxidants as for natural rubber latex can be used but at higher quantity because the level of anti-oxidant present in the raw Cariflex IR0401(B) latex polymer is very low. Phenol type anti-oxidants are typically used.

Sulphur, zinc oxide, accelerators and solid antioxidant dispersions can be bought as such or can be prepared by oneself. The advantages of the ready to use ingredients are obvious (no search for stabilizers, no milling/emulsification). The advantage of the second approach may be the optimal selection of the stabilizing components (and price of the ingredients).

A formulation containing minimum amounts of chemicals is shown in Table 6.

Table 6: formulation for an IR0401(B) compound

Ingredient	Amount [phr]
IR0401	100.00
ZnO	0.25
Sulfur	0.64
ZDEC (Zinc diethyl dithiocarbamate)	0.50
DPG (N,N'-Diphenylguanidine)	0.50
Wingstay L*	2.00

* Butylated reaction product of *p*-cresol and dicyclopentadiene

The formulation is chosen such that minimal amounts of chemicals are used, to achieve the right properties after dipping and vulcanization after one day of maturation.

Cariflex IR0401(B) latex polymer can be mixed with other synthetic lattices provided they are anionic or non-ionic stabilized and have a pH above 9. It can be blended with natural rubber latex at any ratio. It is recommended to add first a stabilizer to Cariflex IR0401(B) latex polymer, before adding the natural rubber latex, or another latex type.

Mixing of different batches of IR0401(B) or batches of IR0401 and IR0401B goes without problems. The compounded latex can be stabilized with 0.75 phr sodium caseinate. This can best be added to the latex prior to the addition of the other components. Stabilization can also be achieved by 0.4 phr sodium dodecyl sulfate. The concentration of other ingredients in Table 6 should then be increased to obtain similar mechanical properties.

After mixing the latex with the compounding chemicals, the mixture is pre-vulcanized (matured), before use. Typically, storage times of 2-16 hours at ambient temperature, is sufficient for adequate dispersion of the compounding ingredients. Besides, maturation allows time for any air bubbles in the mix to rise to the surface. The mix should be gently stirred during maturation to prevent creaming and skin formation. After maturation the mixture may be sieved.

Maturation can be followed by measuring the degree of vulcanization of pre-vulcanized natural rubber latex¹⁵:

1. The chloroform-coagulation test. In this test the latex is stirred with a certain amount of chloroform, and the degree of vulcanization is then judged from the appearance of the coagulum which forms. The method is rapid.
2. The equilibrium-swelling test. The degree of vulcanization is judged from the extent of equilibrium swelling of films dried down from the latex, the swelling being carried out under controlled conditions using a suitable rubber solvent (such as toluene). The procedure is time consuming.
3. The relaxed-modulus test. Again, a thin film is cast from the latex. The degree of vulcanization is determined from the relaxed modulus at 100% extension (MR 100) of a test piece cut from films dried down from the latex. The method is time consuming.
4. The prevulcanizate relaxed-modulus (PRM) test. The degree of vulcanization is again judged from the relaxed modulus at 100% extension (MR100) of films dried down from the latex. The dried thin film is dried at elevated temperature and then rolled into a ring. The procedure can be carried out within 10 minutes

When the compounded latex is matured to a suitable degree, dipping can begin. Dipping time of the former containing the dried coagulant, depends on the concentration of the compounded latex, and the required thickness of the dipped good. This was described above. Usually, the dipped, latex covered former is then heated for a short time (e.g. one minute) to give some strength to the film. The next step may be a leaching step, to remove water-soluble material from the film. The former is held in a water bath. Residence time and temperature of the water bath must be optimized for the specific formulation. The next step is the actual curing of the film on the former. The time and temperature at which this step is carried out must be optimized for the specific application. Examples of how these parameters influence mechanical properties are given in Figure 9.



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