Midas

MARGINAL LANDS, INDUSTRIAL CROPS
AND INNOVATIVE BIO-BASED VALUE CHAINS

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Guayule latex production and validation gloves manufacturing.

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Executive summary

The first guayule latex production has been realized using the existing GuaTecs's water-based extraction pilot, with the guayule biomass harvest from plots planted two years before launch of MIDAS project. To accelerate the development process, GuaTecs implemented in its laboratory under the MIDAS project internalised the mechanical and chemical stability evaluations. This allowed us to investigate several extraction parameters and additives; numerous latex samples were obtained before achieving mechanical stability and chemical stability targets.

The QUALITY of guayule latex produced by GuaTecs is now validated at small scale for glove manufacture.

While discussing with glove companies (Piercan and others) it appeared that they need tonnes, not tens of kilograms as initially planned, to make straightforward final sound evaluations before launching industrial production. Thus, they postponed final glove trials until the demo-scale pilot unit will be funded and constructed. Therefore, GuaTecs decided to focus now on smaller markets, but with even higher values, as medical devices and others.

The VOLUME of samples is now being increased, through improving the production capacity of the existing pilot unit, passing from 40% to 80% based on polyisoprene recovered in the final "cleaned" latex.

Chapter 1: guayule latex sampling

The first guayule latex production has been realized on the GuaTecs's water-based extraction pilot, with the guayule biomass harvested from plots planted two years before launch of MIDAS project.

GuaTecs's guayule field and latex

GuaTecs's extraction pilot

The quality parameters depend on each specific use. In the present case of glove manufacturing the latex must display a high mechanical stability, but a low stability to calcium ions (see table). The requirements for guayule latex for glove manufacture correspond to the ASTM D 1076, category 4.

D1076 – 21

TABLE 1 Requirements for Specified Latex Categories

	Category 1	Category 2	Category 3	Category 4	Category 5
Total solids, min,%	61.3	66.0	61.3	44.0	61.3
Dry rubber content (DRC),[A] min, %	59.8	64.0	59.8	42.0	60.8
Total solids content minus dry rubber content, max,%	2.0	2.0	2.0	2.0	0.5
Protein content (µg/g dw latex)					
Total protein by D5712				200 max	
Hevea antigenic protein by D6499				None detectable	
Total alkalinity calculated as ammonia, as % latex	0.60 min	0.55 min	0.29 max	0.60 min	0.60 min
Or: total alkalinity calculated as KOH, as % latex				0.10 min	
Sludge content, max, %	0.10	0.10	0.10	0.10	0.1
Coagulum content, max, %	0.050	0.050	0.050	0.050	0.05
KOH number, max[B]	0.80	0.80	0.80	0.80	0.8
Mechanical stability, s, min	650	650	650	90	650
Copper content, max, % of total solids	0.0008	0.0008	0.0008	0.0008	0.0008
Manganese content, max, % of total solids	0.0008	0.0008	0.0008	0.0008	0.0008
Color on visual inspection		no pronounced blue or gray[C]			
Odor after neutralization with boric acid		no putrefactive odor			

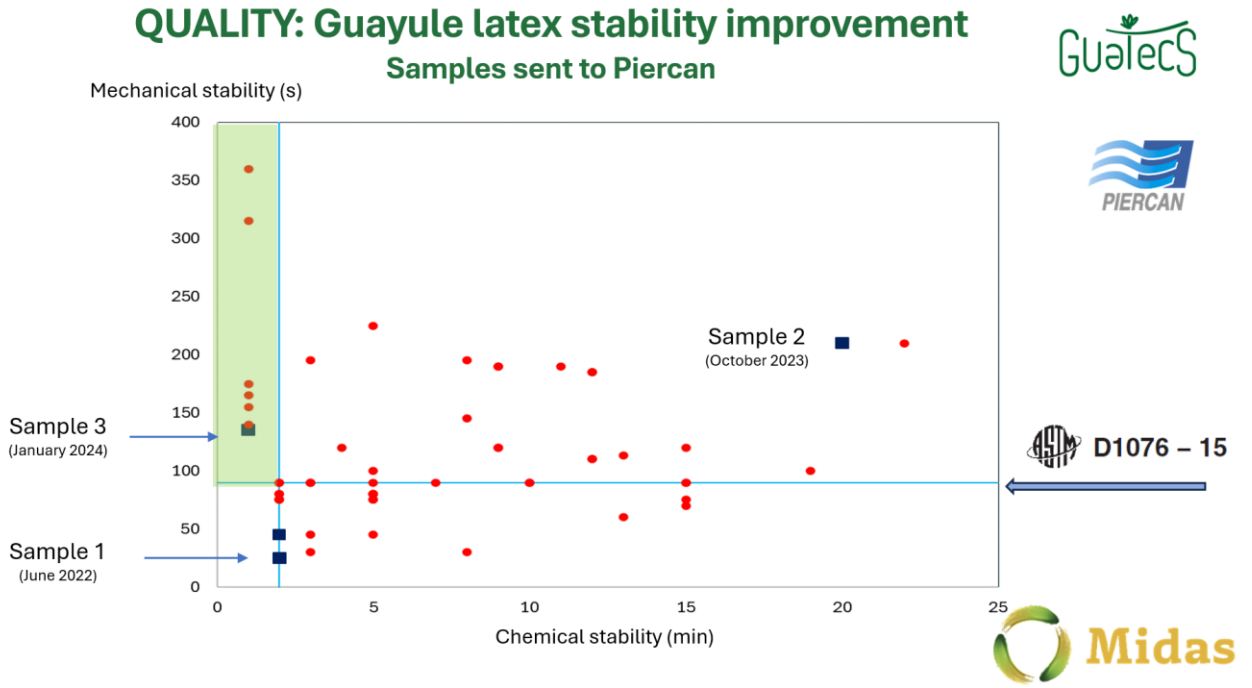
To accelerate the development process, GuaTecs internalised the mechanical and chemical stability evaluations instead of sending dozens of samples to Piercan.

The mechanical stability of latex is the ability to maintain its structural integrity and resist deformation or breakdown when subjected to mechanical forces (stirring, agitation, shearing, or other mechanical stresses). To evaluate this stability, a high-speed mechanical agitation was imposed on the concentrated latex until the first appearance of small pieces of coagulated rubber. The mechanical stability value for a latex is expressed as the number of seconds elapsed from the start of the test to the end point. The target is 90 seconds minimum.

Chemical stability of latex is related to the coagulation of the latex by divalent cations for the production of objects by dipping a mold into the latex. Calcium nitrate is commonly used as a coagulant in this process. To evaluate this: a mold was preheated at 60°C and dipped into a calcium nitrate solution, the mold dried at room temperature for one minute, and was dipped in the latex for one minute. The chemical stability value for a latex is expressed as the number of minutes elapsed from the mold's exit and the complete coagulation of the film. The target is 2 minutes maximum.

The first latex sample produced under MIDAS project was sent to Piercan for quality evaluation for glove application. Mechanical stability and calcium stability have been evaluated:

- Measured mechanical stability of sample 1 latex was 20 seconds. It needed to be improved to allow the latex affording pumping during the glove manufacturing process. The stability target under specific testing conditions should reach at least 90 seconds.
- Measured chemical stability to calcium of the sample 1 latex was found to be 2 minutes, which is too high, the target being 1 minute maximum; indeed, it had to be lowered to increase the coagulation speed of the film and keep the productivity of the glove manufacturing line.



The last guayule latex sample (sample 3) has been successfully evaluated by Piercan.

The wide diversity among the series (more than 40) of obtained and tested latex samples is shown in the figure displaying both the measured stability for each sample. The target sample should be located within the green zone. For example, sample 2 was by far chemically too stable.

Then by continuing to investigate the effect of several process parameters and formulation additives thanks to the expertise of GuaTecs we could achieve the Chemical Stability target while maintaining the mechanical stability at the right value; see sample 3, at 1 minutes and 130 seconds, respectively.

To achieve these goals, it was necessary (I) to act on extraction and cleaning process parameters in order to play with the content of non-polyisoprene components which are extracted together with the polymer, and (ii) to fulfil several requirements, like safety for glove users or manufacturing cost; this strongly complicated the optimization procedure.

Chapter 2: guayule latex quality

The QUALITY for glove production is now validated.

These table shows advantages and drawbacks of the guayule latex in comparison to the hevea latex and also to synthetic latex in the glove application sector. Actually, guayule latex optimized under MIDAS project meets both non-allergenic and stability requirements contrary to hevea or synthetic latex which can meet only one. In addition, the sustainability aspect plays also in favour of the guayule latex which is extracted with water without any chemical solvent.

The sole yet unknown parameter in the table is the production cost, but as can be seen it could be up to the cost of the synthetic latex.

Polyisoprene lattices	hevea	guayule	synthetic
Allergenic aspect	--	++	-
Mechanical aspect	+	++	++
Supply chain sustainability	+/-	++	--
Price (€/kg)	1,5		15

In addition to this technical & economic evaluation with Piercan end-user contribution, a collaboration with Solar Impulse Foundation has been launched to evaluate the environmental & societal values of our guayule latex. The conclusion is that GuaTecs guayule latex has been labelled as an efficient solution, good for the planet, people, and business.

Chapter 3: next steps

In our first analysis, glove application was identified as the application with the smaller market size and the higher value in the new agro-industrial guayule chain. But glove companies (Piercan and others) need tonnes and not tens of kilograms to make final evaluations before launching industrial production. Thus, the glove trials are postponed until the demo scale extraction pilot be funded and constructed, possibly after end of project.

Nevertheless, this work has opened the way to further testing towards the sector of allergy-free gloves.

Thus, GuaTecs decided to focus now on smaller markets, with even higher value as medical devices and others, and requiring lower samples volumes for latex testing before industrial production as shown in the hereafter figure.

Anyhow, as initially planned the production capacity has to be increased. This was done by first having a better knowledge of the colloidal behavior of the latex along the various processing steps (already completed), and then upgrading the present pilot-scale exaction unit by adding new devices.

Being synthezied and entrapped within the cytoplasm of specialized cells in the branches of the guayule bush, the polyisoprene cannot be extracted by simply letting it flowing out as a latex like for the Hevea tree, neither by pressing. Polyisoprene is not under a latex form inside laticifer channels in the case of guayule. At this point we must recall that, although polyisoprene be insoluble in water, water is necessary to recover it under the targeted latex form (polymer particles dispersed in a water phase).

Indeed, the applied patented process -to which GuaTecs founders contributed- is based on a peculiar grinding of guayule biomass in a water phase. Heating is not allowed to avoid thermal coagulation of polyisoprene particles.

Therefore, extracting all polyisoprene would imply breaking the wall of all concerned cells which is practically impossible in a very short time at ambient temperature. Moreover, polyisoprene particles are potentially subjected to coagulation during extraction (high speed shearing), purification, and concentration steps.

Because all above constraints, not only all polyisoprene is not fully extracted from the biomass, but also extracted macromolecules are lost during further processing steps.

The physical and chemical phenomena involved in the course of water-based polyispprene extraction process have been investigated by GuaTecs under the frame a PhD work. Polyisoprene particles inside cells are surrounded (protected) by a complex shell of proteins and lipids, which govern their colloidal stability. This shell must be preserved during the extraction process, whereas the controlled aggregation of natural rubber particles in the latex is fundamental to the production of rubber materials.

The colloidal behavior is influenced by numerous physico-chemical factors (pH, ionic strength, divalent cation binding). A range of produced guayule latex samples in terms of plant variety, and extraction process, have been studied, each with their unique composition and colloidal properties. The experimental approach spans a broad range of particle volume fractions, from extremely dilute suspensions to highly concentrated ones, offering a comprehensive and complementary view of the system. While considerable knowledge exists about (Hevea) latex behavior in dilute systems, our findings address a critical gap in understanding the aggregation dynamics in highly concentrated suspensions, particularly under the influence of cations. Key experimental tools, including light scattering, stability diagrams, and creaming monitoring, were employed to investigate the onset of aggregation under varying conditions. Distinct mechanisms of latex response to cations and their influence on the suspension's creaming kinetics were identified. These findings shed light on the early stages of cation-induced aggregation and provide insights into the underlying mechanisms.

These results open new perspectives for the industrial optimization of guayule latex stability by fine-tuning the particle shell composition, offering a path to improve latex extraction process and the properties of rubber-based materials

Having in mind above fundamental results, GuaTecs evaluated several routes and identified different ways to increase the extraction yield of polyisoprene. Optimizing the equipment efficiency, process cycle improvements, and water flow optimization (recycling), increased the yield by ~20%, 10% and 10% respectively. This ongoing work lets expecting an overall yield of at least 70 %, compared to the usually obtained 40%.

In addition, other limiting steps are also being improved: adapting a harvesting machine at very small scale (instead of the current manual harvesting with a chain saw), and biomass milling.

The consortium