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Hevea Bralizens Is Natural Rubber:– A Review on the Macro- and Micro-Structure of the Hevea Hydrocarbon Chain in its Natural and Chemical Forms, Especially in the Mma Grafted Hevea Natural Rubbers By IR Spectroscopy Including the ATR FTIR Spectral Analyses

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ABSTRACT

The Hevea natural rubber molecule can be considered to be polyisoprene, in which individual isoprene molecules are joined by 1, 4-addition. It is possible to represent this polyisoprene in terms of the cis and Trans isomeric forms. Natural rubber is obtained commercially from the sap of trees called Hevea Brasilensis. These trees yield latex containing approximately 35% rubber hydrocarbon and 60 % of the aqueous fluid and 4-5 % of the proteineous components, lipids, fatty acids and resinous residents. The hydrocarbon polymer consists of 97% cis-1, 4 units, 1% trans-1, 4-units and 3% 3, 4 units in a head-to-tail structure. It was established and reported that the average molecular weight of cis-1, 4-polyisoprene is approximately equal to 38 000 and nearly 558 isoprene units are there on average in a given polymer chain.

It was discussed that the infrared spectra and structures of the Hevea polyisoprene would be changed due to chemical reactions during the production /process like halogenations, grafting or even chemical substitution in the sulphur vulcanization. In addition to the tentative assignments, the R & D chemists also pointed out the possibility of the occurrence of the vibration modes with small differences in spectral bands for the interpretation of spectral bands produced by chemical reaction and modification during the process.

Thus, the manufacturing process of the MG50 latex , where 50 % 0f the methyl methacrylate (MMA) was grafted, the main backbone of the Hevea polyisoprenoid chains remained almost the same, except where the molecular sites and configurations are chemically modified in terms of carboxylate, hydroxyl groups, and some minor cis- and trans- configuration changes. The individual spectral vibration or bending modes of molecular structure would be changed, but a little or without any significance, and the difference of the spectral bands are within the ± 2 cm$^{-1.}$ Any presence of the inorganic or metallic ions would not change these spectral mode significantly either, as shown in the IR spectral modes of the present MG50 latex.

The MMG grafted hydrocarbon poly-isoprenoid chains are stereo-structurally in tack, except changes in the chemical grafting occurred where the isomers configurations also evolved and changes slight, and some isomers are " destroyed " through the chemical reaction like carboxylate, (- C O – OH) or hydroxide (-OH) groupings, and some these are overlapped or overshadowed, for example, the sharp peak at 1274 cm^{-1} with symmetrical stretching is assigned to the C =O bond , and also was reported that 1387, 1278 and 987 cm^{-1} as O – CH .It is interesting to note that the peaks 835 cm^{-1} and 1720 cm^{-1} , that are related to the – C = O or – CHO groups , or corresponding to carbonyl, aldehyde , ketone , carboxylic acid, or esters groups .

Keywords: *Hevea NR, macro and micro-structures of polyisoprene isomers, ATR and IR Spectral bands; Z-1, 4, E-1, 4, 1.2, 3, 4 isomers; methyl methacrylate MMA, MG50 Latex, MG30 rubber, non-rubber components,*

SPECTRAL ANALYSES

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Hevea Brazilensis Plants from Brazil

In the historical events, the Hevea Brazilensis plants were imported from the London Kew Garden, then thro' Kew Garden of Colombo and

Singapore to Malaysia in 1884. The ten trees arrived at the Kuala Kangsar Agriculture Station and from these ten trees, through the natural and hybrid bud-grafting, cross-pollination and clonal selections, etc. there are over 100 species today in Malaysia and over 300 clones in the ASEAN countries being created. The rubber latex is produced in the leaves through photosynthesis and then transported to the stem area that lied between the cambium and the bark. By puncturing the bark through the cork region, and breaking the latex cells, the latex oozes out and by the stem pressure, it is forced and flows out and be collected in a conical cylindrical cup. In a normal situation, it takes 3-5 hours of flow, and then it stops flowing. This is caused by the drop in the stem pressure and also caused by the slow coagulation of the latex by the bark that is being petrified by the bacterial action.

Hevea Bralizensis Natural Rubber

Natural rubber is harvested from a rubber plant called Hevea Bralizensis which is originated from Brazil, and in the field latex stage, it has 62 -65 % of water and the rest composes of 29-33 % of pure rubber and the rest known as non-rubber consisting of , lipids, fatty acids, sugars, and proteins. These non-rubber components, especially the fatty acids present that assist to stabilize the latex**.** The rubber latex is a complex natural composite of pure hydrocarbon-polyisoprene (93-95%), proteins, phospholipids (1-1.5 %), sugar (1.5 -2 %), fatty acids (1-2 %) and its density is 0.92. It is important that NR has also naturally proteineous products, sugar, carbohydrate, sterols and phospholipids which are parts of the naturally occurring antioxidants. It is the present of these naturally occurred antioxidant that make the natural rubber unique, and cannot be imitated by the synthetic process in the production of synthetic polyisoprene.

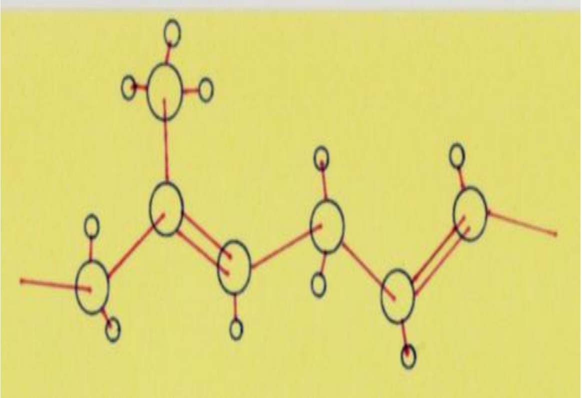

Figure1. *Isoprene unit*

Micro Structures of the Hevea Rubber Hydrocarbon Chains

Apart from these natural occurring products, the natural rubber has a very long molecular chain, that is, at least 1,000,000 unit equivalents to the hydrogen molecule, while the common synthetic rubber has only 300,000-350,000 units. Its macro and micro-structures and their nature were most studied, using almost every other scientific methods and techniques available and known to measure their chemical and molecular constituents, their functionality in the micro- and bulk states.

Among many scientific and technical methods used in the study of these micro-structures, the Raman and infrared spectroscopy are commonly used and currently ATR FTIR techniques is used, and offer unique information about a number of polymer characteristics such as identification, composition, structural information, and configuration. Over the years, spectroscopic studies of polyisoprene have been made by a number of workers [1–7]. The increasing interest in the vibration spectra of cis-1, 4-polyisoprene is due to its vast industrial applications. The Hevea rubber molecule can be considered to be polyisoprenoid hydrocarbon, in which individual isoprene molecules are joined by 1, 4-addition. It is possible to represent this polyisoprene in cis and Trans isomeric forms. Natural rubber is polyisoprene [8]. It is obtained commercially from the sap of Hevea Brasilensis trees. These trees yield latex containing approximately 35% rubber hydrocarbon and this hydrocarbon polymer consists of 97% cis-1, 4 units,1% trans-1,4-units and 3% 3, 4 units in a head-to-tail structure.

The figure shows the chemical structures. The NR hydrocarbon chain configuration is head to tail entanglement which indicates about 3% Trans-units per chain. The structure of natural rubber has been Investigated for over a hundred years; it was only after 1920, however that the chemical structure was elucidated. The average molecular weight of cis-1, 4-polyisoprene is approximately equal to 38 000 and nearly 558 isoprene Units are there on average in a given polymer chain. are isomers of the isoprene units [1, 2**]**Further it was discovered that the hydrocarbon of the Hevea Brazilensis has made up of mainly the Z-1,4 isoprene units, and its IR spectra satirically are characterized by the spectral bands at 1130 cm^{-1}[v(C–C)] and 838 cm^{-1} [ɣ(=C)] ; whereas the Gutta Percha and Balata produce mainly the E-1-4 isoprene units, and in its FTIR spectra, it was showed that stable spectra lines at the 1050, 1030, 990, 863, and 418 cm^{-1}.The bands of the 1,2- isoprene and 3, 4–isoprene are at 890 cm^{-1}, as well as 1003 and 910 cm^{-1}. The summary of these spectral bands are as shown in the table 1, by Saunder & Smith, et al) [1, 2] below.

Table1. *Isomeric isoprene units*

Z - 1, 4	**E – 1,4**
4064	2973
2963	1385
1377	1325
1130*	1150*
837**	843**
762	980
742	600 (interference as hi – Z –content)
572	
3, 4 isoprene unit	**1, 2 isoprene unit**
3070 (at hi-3.4 content) 910 1780	

**Only in the Hevea Balata blends, or in poly-isoprene block co-polymers*

***Only one band at 840 cm^{-1} in mixtures, as reported according to Saunders and Smith, etal*

The prominent features of the isomeric units as observed are

- There are 77 % of the isomeric units, E -1, 4; 12 % Z-1, 4, and 5 -6 % are in the 1, 2 and 3, 4 isoprene isomer units.
- The Hevea hydrocarbon chain mainly characterized by spectral band 1130 cm $^{-1}$, and 838 cm^{-1}, others present are 1740, 1705, 1630, 1535, 1600, 1650, 1710, and 720 cm^{-1}.
- Whereas the E -1, 4 configuration has 1050, 1030, 990, 863, 418 cm^{-1}, and
- The 3,4 and 1,2 sequences/configuration show the spectral bands 890, 1003, 910, and 760 cm^{-1}, though the vinylidene spectral also the same spectral band 890 cm $^{-1 -.}$.
- The Hevea NR has a variety of resins and proteins, and fatty acids and ester. These with its own spectral lines tend to interfere with the polyisoprenoid structures. For example, the ester and acid grouping – their spectral bands like 1740 and 1705, and the protein group 1630 and 1535 cm $^{-1,}$ interfere with the normal polyisoprenoid groups, thus, they are not shown in the over-all spectra lines and defused.

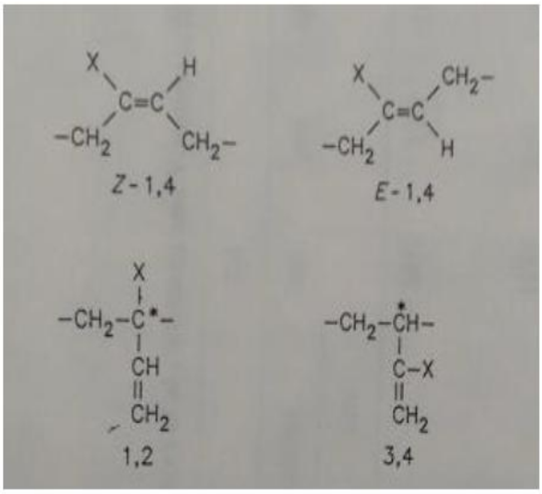

Figure2. Isoprene isomers

All the data and spectral bands mentioned above are important, and would be used to discuss and verify the latest findings in many new technical papers related to the MMA grafted natural rubber.

Nallasamy and S Mohan [8, 9] has further also established and reported the structural parameters of the cis-isoprene in terms of the atomic lengths and the atom-to-atom space co-ordinate. They are recorded:

Table2. *Inter atomic to atomic space and angle of atomic structure*

(= C – H) length is 1.09 Angstrom	**the inter - atomic special angles ∠ are**
(- C – H) length is 1.10 A	∠ (-C –C = C -) in degree 125
(- C = C -) length is 1.34 A	∠ (-C–C– C- in degree 109.5
(= C- C-) length is 1.54 A	∠ (H – C – H in degree 109.5
(- C –C -) length is 1.54	∠ (C = C – H in degree 120

In fact, there were 22 inter-atom co-coordinator spaces were reported in the paper, and they even calculated the frequency and the potential energy distribution (PED) for each vibration assignment.

For example; Symmetrical bands such as C = C, or C–C, for example, the Raman IR spectral band at 1665 cm^{-1} and the IR counterpart identified as 1667 cm^{-1}. The other three skeletal modes contributing to the C – C stretching is assigned to the spectral band at 1130, 943, 925 $cm^{-1.}$

In the Methyl Ch3 Modes

In their observation, in the strong Raman and IR bands, observed at 2970 and 2964 cm^{-1} respectively, that belongs to the CH3 asymmetric stretching modes, and they quite agree with the calculated numbers 2961 and 2957 $cm^{-1.}$

The frequency of the CH3 "rocking vibration "of the unsaturated hydrocarbons occurred at the 950-1150 cm^{-1} region, that is, at 1104 cm^{-1}, and the calculated is at 1031 cm^{-1} All the above reported differences in terms of the vibration frequency for each mode would further discussed in the subsequent reports and observed spectral line, and their changes due to the environmental changes or influences. In fact, there are/ were exact location of the structural band for each vibration or bending motions in a molecular structure. It was reported that some of them are a range of even ± 5 cm^{-1}

Chemical Modification of Hevea Hevea Brazilensis Natural Rubber

The natural rubber derived from the Hevea Brazilensis is a green material which is organic, natural and abundant, and renewable. The various aspects and methods of chemical modification of natural rubber (NR) remains as one of the alchemists in the development of improved NR.

Such a modification can be achieved by just blending suitable chemical additives in NR, and by chemical reaction in the NR phase, through a solid state, latex phase, and even in the solution state or surface modifications.

These modifications have improved the normal and elastic behavior of NR. The readily reactive nature of the Hevea hydrocarbon chains, especially the isoprenoid units which are reactive and their double carbon-carbon bond at the 5th carbon ensures an easy chemical addition. Without detailed discussion of the chemical reactions, the chemical medication of the NR could achieve in the following:

- Addition of halogens and hydro-halogenaton
- Chemical substitution
- Free radical or irradiation
- Grafting co-polymerization
- "ene "reaction mechanism

In the early 19th century many chemical modifications of NR were successfully made at the laboratory scales. These were academic interests and achievements. However, some had commercial needs and viability, e.g., chlorinated NR [12, 13, 14], and cyclised NR [2, 15], were useful materials. During and after the World War II, with the discovery of synthetic rubbers, plastics, polymeric resins and oligomers, these modified NR's were replaced by the later synthetic materials which are produced more cheaply.

Even today, NR remains one of the most important, industrial and sustainable materials. It is abundant and renewable. NR has balanced, physical and chemical properties that no synthetic rubbers can match. It is highly elastic, and has high tear resistance, high tensile strength, good wear and abrasion, high flex and good fatigue, and dynamic properties.

It is easy to process and good tack for fabrication to many green components, and finally easy and cheap to cure or vulcanization using the sulphur curing systems. However, NR has some deficiencies like poor oil- resistant, weathering and heated environment. All these could be overcome by the chemical modifications.

The improvement include non-tackiness, hardening effect, improved in rubber-to-metal bonding, more flame resistance, more heat resistance, improved gas/air impermeability, better dimension and shape tolerance and even improved oil resistance. Some surface modification even has potential UV photo-lithographic effects. However, many of these modifications have not been fully exploited and commercialized. Other than the cyclised NR and chlorinated NR, one other modified NR's is the methyl methacrylate (MMA) co-grafted NR to the extent that 30 – 49 % achieved, or what are known as MG30 and MG49 which Malaysia is the only country producing these materials.

Methyl Methacrylate (Mma) Grafted Hevea Nr, Mg 50 Latex

In this paper, a commercial method of the production of the MG 50 latex was established more than many decades. The process briefly involved a methyl methacrylate (MMA) monomer emulsion contain a hydro peroxide (producing free radical reaction centers is mixed with ammoniated field latex where a mine added to initiate the chemical reaction in the latex molecules they acts for the formation of the polymerized chain.

On the following day after the polymerization competed, the dispersion of the anti-gradient such as like anti-oxidant is added a protective rubber chemical. The final MG50 Latex was then stabilized, and in this case, a special ant-ozonant is also added, making the MG 50 latex weather resistant and also more atmospheric ozone resistant. The author likes to discuss the micro-structures of the MMA grafted Hevea natural rubber. The MG30 and MG50 are mostly extensively developed and used in the industrial market, and also researched by both the R&D chemists and industrialists. The records and results of the IR, FTIR, Raman IR and the more recent ATR FTIR spectral lines/bands reported are very diverse, and might not correlated to the nature of Hevea natural rubber where the MMA grafted. Many research workers claimed that what the spectral lines/bands were due to the present and effects of the inorganic fillers added, sulphur vulcanization, due to the environmental factors, or due to the chemical treatment like irradiation.

Observation of the Early Work on the MMA Grafted Hevea Natural Rubber

Early IR, FTIR and ATR FTIR Reports on MMA grafted- Hevea NR In the PMMA, it was reported that the FTR spectral bands due to the saturated polymeric ester, C = O, its systematic stretching frequency gives rise to a very strong peak of 1732 $cm^{-1,}$ and other vibration peaks of 1387, 1274, and 987 cm^{-1} that is assigned to the O-CH, deformation, C – OH and C – O –C symmetric stretching of the PMMA. In the work of the MG30 based electrolyte where a number of inorganic fillers were incorporated, the FTIR spectral bands changes, or shifted. They were observed as follows: MG30 –Li trifate has 766, 1033, 1182, and 1260 $cm^{-1,}$ and In the MG30 – Al_2SO3 , the peaks are 1040, 1373, 1450, 2841 and 2903 cm^{-1}., and in the bands in the spectrum obtained are 974, 1070, 1172, 1396, 1841, 1274, and 1803 cm^{-1}. The small changes in the spectral band observed was explained by and due to the coordination of the metal ions like the $Li^+ \leftarrow - O = C$ (the carbonyl group), and new peaks like 1779 and 1809 cm^{-1} are observed responsible to this coordination phenomenon. In the present work, where the MG50 latex in a semi-fluid form were analyzed using the Bruker ATR FTIR spectrometer. The ATR FTIR spectrum is shown in the (figure 3) below.

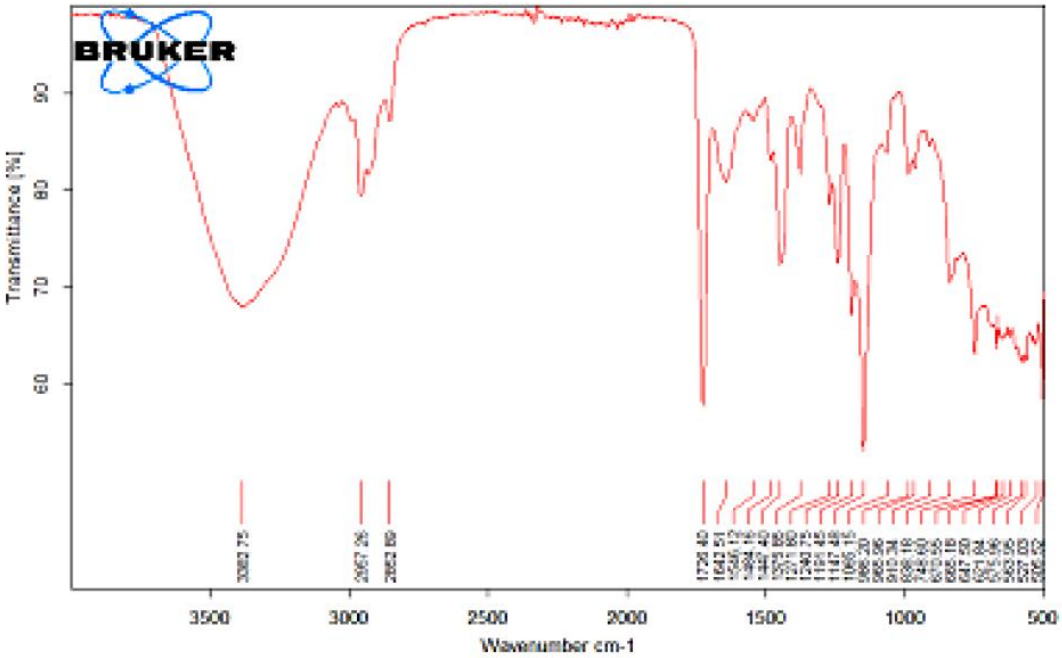

Table3. *It's observed spectral bands*

Of MG50 latex ATR FTIR IR						
are (in terms of cm $^{-1}$)	**Hevea**	**NR**	**PMMA**	**MG30**	**+ Li-salts**	**EC****
3382,	3282.8	3037	2962	vulca*		
2957.3	2918.8	2960	2890	1803	1803	1804
2852.9,		2853	1778	1774		1774
1726.4,.. – C=C- bending	1725	2715				
1642.5, .. 3,4 - isomer	1625.7	1736				
1484.2,	1433.1	1663	1447	1481		1481
1447.7		1448				
1375.8, ..3,4- isomer	1373.4	1376	1387	1375		1396
1272.8,	1260.5	1309	1274			
1240.7,..1,4-isomer	1260.5	1231	1234		1230/1260	
1191.4,	1090.4	1095			1182	1172
1065.1,	1018.1	1038		1076	1033	1076
986.2, ,.. 1,4-isomers	987	872	984	974		
91.03						
838.2,….1,4-isomers	835.6					
748.8,	712.1	740	718-720	766	766	
670.6,	670.7			670		
665.4,	665.2			665		
647.5						
621.8						
575.9,	534.1					
563.9,.. 3,4- isomer						
527.1,	534.1					
505.5	517.6					

MT30 vulcanized S/Accel systemEC- electrolyte system, Li-salt i.e. Lithium trisulsphate, PMMA - Polymethyl methacrylate*

The spectral bands, 3383.2 and 2957.3 cm$^{-1,}$ are due to the water / moisture on the surface of the MG50 latex being semi-dried. Firstly we compare the range of the spectral bands of the MG50 latex and the Hevea latex (atrftir). Traditionally the Hevea NR has the isoprenoid isomers 1,4; 1,2; 3,4 configurations, and their percentages are 97 % for the 1, 4 isomer, and 2, 5-3.0 1, 2 – and 3, 4 – isomers.

Through the chemical reaction and changes in the structural configurations, the bond – C = C vibration mode at 1274 cm^{-1} shifted to 1276.2 cm^{-1}, and the bond – C H–CH3 bending mode remains almost the same, 1375.8 cm^{-1} again 1373.4 and 1396 cm^{-1} in the ART FTIR and IR spectra of the Hevea NR respectively.

These are minor differences would not affect the spectral identifications as reported by Nallasamy & Mohan. However, in this rep\port the configuration of the MG50 latex hydrocarbon structures, as it shows the present of the 1, 4 and 3,4–isomers, and possibly some trans-isoprenoid isomer units. The molecular activities of MG50 latex, PMMA, and MG30 rubber are similar as shown in spectral bands such as:1726 (-C = C, bending mode in PMMA, MG50, and even MG30O); 1642 (3, 4-isomer; also Sp2 of –C=C); 1447 (appears in MG30 and PMMA); 1373 (3-4 isomer; also appears in MG30);1272 (-C=C vibration, also appears in PMMA), 986 (1.4-isomer; -O-CH asymmetric vibration, also seen as –C-O-C in PMMA), 838 (1, 4-isomer,-C=C bending mode); 575, also seen in MG30); 670 (uniqueness as seen on MG30, and also to be a vinyl/alkenes substitution).

All bands listed above are in cm^{-1} any changes observed in the MG50 latex and MG30 are due to the present of the inorganic/captions that could influence and shift the spectral bands. These were discussed in the respective papers [7, 17, and 19].

In the production or process where the MMA is grafted, the hydrocarbon poly-isoprenoid chains are stereo-structurally changes as the chemical grafting takes place, and also the isomers configurations also evolved and changes, some isomers are "destroyed "through the chemical reaction like carboxyl ate, (-CO–OH) or hydroxide (-OH) groupings, and some these are overlapped or overshadowed, for example, the sharp peak at 1274 cm^{-1} with symmetrical stretching is assigned to the C =O bond and also was reported that 1387, 1278 and 987 cm^{-1} as O–CH in MG30 by K Kumutha et al [17]. It is interesting to note that the peaks 835 cm^{-1} and 1720 cm^{-1}, that are related to the –C=O or –CHO groups, or corresponding to carbonyl, aldehyde, ketone, carboxylic acid, or esters groups [15]

SUMMARY

The rubber molecule can be considered to be polyisoprene, in which individual isoprene molecules are joined by 1, 4-addition. It is possible to represent this polyisoprene in cis and Trans isomeric forms. Natural rubber is polyisoprene [8]. It is obtained commercially from the sap of trees called Hevea Brasilensis. These trees yield a latex containing approximately 35% rubber hydrocarbon and *this* hydrocarbon polymer consists of 97% cis-1, 4units, 1% trans-1, 4-units and 3% 3,4 units in a head-to-tail structure [9].

It was established and reported that the average molecular weight of cis-1, 4-polyisoprene is approximately equal to 38000 and nearly 558 isoprene units are there on average in a given polymer chain. It was reported and discussed the infrared spectra and structures of the Hevea polyisoprene change due to the chemical reaction during the production/process like halogenations, Grafting or even chemical substitution in the sulphur vulcanization.

In addition to the tentative assignments, they also pointed out the possibility of making use of the vibration mode for the interpretation of spectral changes produced by the production process.

Thus, the manufacturing process of the MG50 latex , where 50 % 0f the methyl methacrylate (MMA) was grafted, the main backbone of the Hevea polyisoprenoid chains remained almost the same, except where the molecular sites and configurations are chemically modified in terms of carboxyl ate, hydroxyl groups, and some minor cis- and trans- configuration changes, and individual spectral vibration or bending modes of molecular structure would change without any significance, and even enhance within the ±2 cm^{-1}.

Any presence of the inorganic or metallic ions would not change these spectral mode significantly either, as show in the spectral modes of the present MG50 latex. The MMG grafted hydrocarbon poly-isoprenoid chains are stereo-structurally in tack, except changes in the chemical grafting occurred. where the isomers configurations also evolved and changes slight, and some isomers are“ destroyed “through the chemical reaction like carboxyl ate, (-C O–OH) or hydroxide (-OH) groupings, and some these are overlapped or overshadowed, for example, the sharp peak at 1274 cm^{-1} with symmetrical stretching is assigned to the C=O bond, and also was reported that 1387, 1278 and 987 cm^{-1} as O– CH.

It is interesting to note that the peaks 835 cm^{-1} and 1720 cm^{-1} , that are related to the – C =O or –CHO groups, or corresponding to carbonyl, aldehyde , ketone , carboxylic acid, or esters groups .

Appendix Preparation and Production of the MG 50 Latex

The process of grafting the MMA onto the Hevea NR involves the addition of the hydro peroxide/redox agents that form free radicals in the rubber molecules; and with the present of an amine, the free radicals act as central points for the polymer side-chains where the polymerization catalysis occurred that initiate homo polymer formation.

In the commercial process, ammoniated NR latex concentrate is added equal volume of water followed by the calculated MMA concentration and dispersion to the required rubber: MMA polymer ratio. The MMA monomer and latex are continuously mixed and stirred until the polymerization is completed, usually within a period of two hours. The ant-degrading like the BHT is added, but in this, case, the phenylediene is added to improve the weather resistance and one-resistance.

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