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Preparation of Basic Magnesium Chloride Whisker and Reinforcement in Chloroprene Rubber (CR322)

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Abstract: basic magnesium chloride whisker was synthetized by using industrial light burned magnesia powder and hydrochloric acid as raw materials, the product formula of $3Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$ were confirmed by XRD testing. After the silane coupling agent modification, the chloroprene rubber(CR322) were prepared according to the formula and the modified basic magnesium chloride was added as different addition amount, the result indicated that the mechanical properties of the CR322 has been obviously improved, as the addition amount of five percent of the modified basic magnesium chloride whisker, the neoprene mechanical properties such as tensile strength and maximum load were increased by 15.36% respectively, and elongation at break was increased by 54.6%, but stress at definite elongation was reduced by 45.57%.
Keywords: basic magnesium chloride, whisker, modification, chloroprene rubber.

Introduction

The alkali type magnesium salt whisker, including basic magnesium chloride whisker, magnesium hydroxide sulfate whisker, basic magnesium carbonate whisker, and magnesium borate whisker can be easily prepared by mild aqueous reaction or thermal reaction[1-5], Alkali type magnesium salt whisker has the characteristics of multistage decomposition dehydration，the dehydration temperature reached up to 450-900°C，the importance to ultimate product dehydration is nature digestible，and even more importantly the product of fibrous structure remain the same after lost water in thermal decomposition，when the fibrous structure of dehydration products in makings gather on the substrate of the composite material，the volatile substances and oxygen transfer were limited. And it was to strengthen the carbonization layer mechanical properties[6,7], the alkali type magnesium salt whiskers were used as flame or fire retardant, reinforcement, antiwear and mechanical performance, viscosity and peel strength and smoke suppression in polyethylene, polypropylene, methyl vinyl silicone rubber, liquid silicone rubber and natural rubber et.al.[8-16]. Neoprene application is very extensive in the field of elastomer such as moulded products，sebific duct, slab rubber, cable belt，conveyor belt material and rubber seals et.al.，neoprene has excellent mechanical strength, high resistance to ozone and weather resistance, good aging resistance, low flammability, good resistance to chemicals, a moderate amount of oil resistance and flame resistance，it can stick to many composite material being formed on the substrate. The application of basic magnesium chloride whisker in neoprene has not been reported, the basic magnesium chloride whisker was synthetized by using industrial light burned magnesia powder and hydrochloric acid as raw materials[17-18], the

chloroprene rubber(CR322) were prepared according to the formula and the modified basic magnesium chloride whisker was added as different addition amount, the mechanical properties of the CR322 such as tensile strength, maximum load, elongation at break and stress at definite elongation were studied in this paper.

Experimental

Industrial light burned magnesia, chlorhydric acid, silane coupling agent (KH-570), acetone were used as raw materials. thermostat water bath with stirrer，drying oven，open mill(XK-160)，no-rotor vulkameter(GT-M2000-A)，press vulcanizer(QLB-50D/Q), tension tester(A1-7000-GD), scanning electron microscope SEM（JEOL JSM-6360LV)，XRD(Bruker D8 Advance)，and infrared spectrometer IR（NEXUS 470） were used as reaction or detection set-up. The industrial light burned powder were slowly added in the HCl solution(6.5mol/L)，the mole ratio of hydrochloric acid with the light burned powder was 0.7:1，a small amount of polyethylene glycol was used as dispersing agent, maintain the reaction temperature at 50℃ for one hour. The insoluble substance can be removed by filter, The filtrate were placed at room temperature for two days, white powdered basic magnesium chloride whisker can be obtained by filtration, and washed by water and ethanol for several times, dried in the oven at 70°C. Then 0.5% of the silane coupling agent was added under mechanical agitation with acetone as the dispersing medium, dried in the oven at 60°C for three days, the surface modification of basic magnesium chloride whisker were prepared by silane coupling agent. According to the formula, CR122 100, zinc oxide 5, stearic acid 0.5, accelerantM 1, sulphur 3, dioctyl phthalate 6, carbon black 20 were formed. And the modified basic magnesium chloride was added as different addition amount from 0 to 30 percent. Neoprene was mixing in a double roller mixing machine for 24h, and then batch-off after mixing. The vulcanization characteristics of mixing rubber were measured by no-rotor rubber vulcanization instrument at vulcanization temperature 170℃ and vulcanizing time 40 minute, The samples was to mold vulcanization on the plate vulcanizing machine according to the measured conditions.

Results and discussion

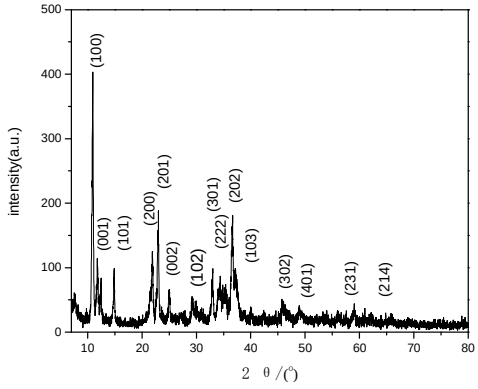

Figure 1: XRD pattern of basic magnesium chloride whisker

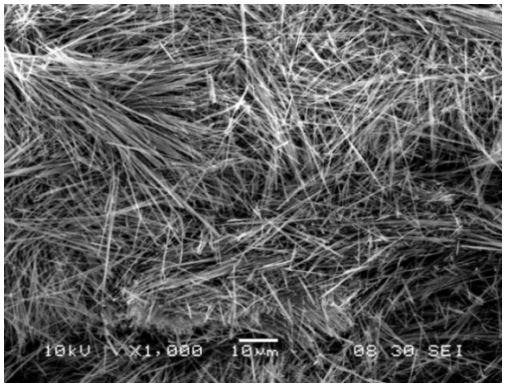

Figure 2: SEM image of basic magnesium chloride whisker

The product of basic magnesium chloride synthesized by different materials molar ratiowere tested by XRD and SEM, the results were showen in the figure 1 and figure 2 respectively，the result indicated that the crystal structure of the product was identical with the standard samples cards（JCPDS 07-0412), and high purity basic magnesium chloride whiskers were obtained in the figure1, the molecular structure was $Mg_2(OH)_3Cl•4H_2O$ or $3Mg(OH)_2·MgCl_2•8H_2O$. it can also be seen that the fiber type basic magnesium chloride whiskers has a good line-type structure, smooth surface and uniform dispersion in the figure 2.

The IR pattern for the original basic magnesium chloride whisker and modified by silane coupling agent were shown in the figure 3. The absorption peak nearby 3443 cm-1 was the O-H stretching vibration peak, and new absorption peak at 3608cm-1 was caused by the -OH stretching vibration characteristic peak of silanol group of silane coupling agent(Si-OH), bounded the silane coupling agent with basic magnesium chloride surface together，and basic magnesium chloride was on the surface of the hydrophilic. SEM image of modified basic magnesium chloride whisker was shown in figure 4. It was clearly seen that the surface of the modified products were enveloping with gray matter, and the diameter of whiskers being increased. It was confirmed that the coupling agent were fixing on the surface of whiskers combining with the IR spectra.

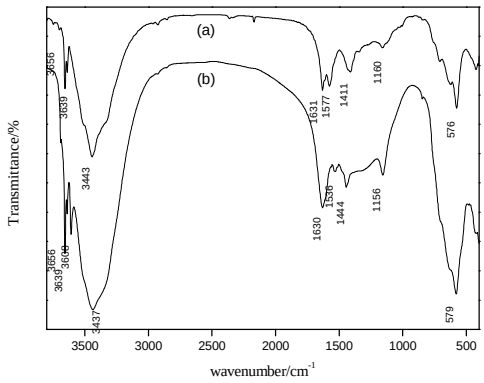

Figure.3: IR pattern of basic magnesium chloride whisker, (a) original, (b) modified

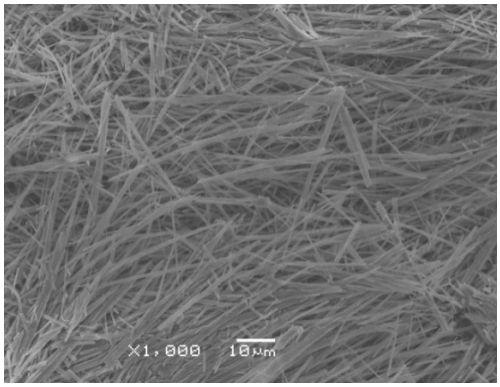

Figure.4: SEM image of modified basic magnesium chloride whisker

The mechanical properties of the neoprene added with different quantity of modified basic magnesium chloride whiskers were tested, and the results for tensile strength, maximum load, stress at definite elongation, and elongation at break were shown in table 1. It can be seen that with adding different quantity of modified basic magnesium chloride whisker, the neoprene with different mechanical properties. When content of modified basic magnesium chloride whisker was five percent, the mechanical properties of the neoprene were improved，such as tensile strength, maximum load were increased by 15.36% respectively，elongation at break was increased by 54.6%，and stress at definite elongation decreased by 45.57%.

Table.1: Result of mechanical property of chloroprene rubber

Percent % (modified product)	Maximum load N	Tensile strength MPa	Stress at definite elongation Kg/mm^2	Elongation at break %
0	225.05	18.75	11.5	396.49
5	259.60	21.63	6.26	612.96
10	243.23	20.27	8.83	482.71
15	232.58	19.38	7.10	563.79
20	233.10	19.42	7.58	572.53
25	250.62	20.89	9.39	521.64
30	237.42	19.78	7.60	573.13

The figure 5 shows the SEM micrographs of the tensile fractured surface of the reinforced neoprene products, The percent of added modified basic magnesium chloride whiskers was from 0 to 30 as a, b, c, d, e, f, and g respectively in the figure5. The micrographs illustrate that the interfacial interactions between the modified basic magnesium chloride whisker and neoprene were significantly enhanced after modification, and played an important role in enhancing mechanical properties.

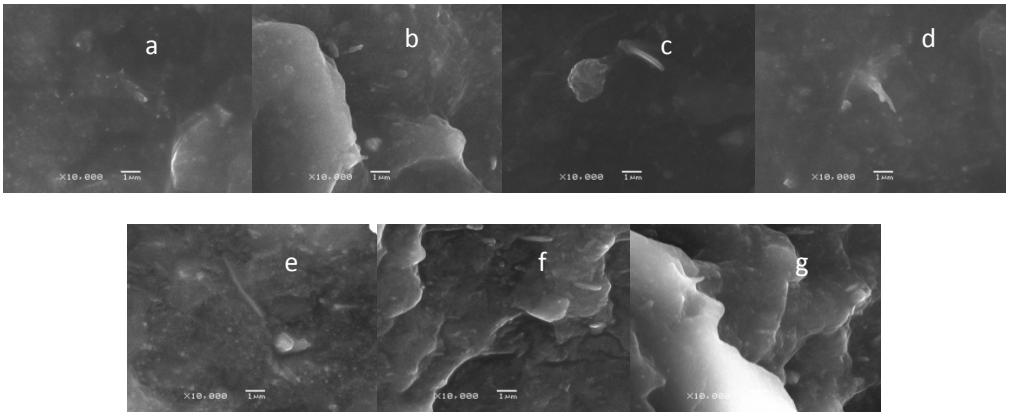

Figure 5 SEM images of chloroprene rubber added modified basic magnesium chloride whisker
Percent (%):a: 0, b: 5, c: 10, d: 15, e: 20, f: 25, g: 30

Conclusion

1 Basic magnesium chloride whisker was synthetized by using industrial light burned magnesia powder and hydrochloric acid as raw materials, the product of $3Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$ were confirmed by XRD.

2 After the silane coupling agent modification, the chloroprene rubber(CR322) were prepared according to the formula and the modified basic magnesium chloride was added as different addition amount.

3 The mechanical property of the CR322 has been obviously improved, as the addition amount of five percent of the modified basic magnesium chloride, the neoprene mechanical properties such as tensile strength and maximum load was increased by 15.36% respectively, and elongation at break was increased by 54.6%, but stress at definite elongation was reduced by 45.57%.

Acknowledgements

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