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Behavior of adhesion forces of the aqueous-based polychloroprene adhesive magnetically conditioned

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ABSTRACT

This research is an innovation proposal for magnetic conditioning of the aqueous-based polychloroprene contact adhesive with the purpose of increasing the adherence capacity between the adhesive and the substrate. Today, the aqueous-based polychloroprene contact adhesive presents an adhesion capacity variation between 1.15 and up to 2.75 kgf/cm². On the other hand, the solvent-based polychloroprene adhesives support average tensions of shearing of 3.8 kgf/cm². To promote an increase in adhesion to the aqueous-based polychloroprene contact adhesive, we formulated one adhesive using a nano-additive, carbon dioxide as catalyst, and a magnetic conditioning process before the phase of application on the substrates. The results obtained show an average increase of 292% in the shearing tension of the adhesive magnetically conditioned when compared with an adhesive of same formulation without the magnetic conditioning and when compared to the commercial aqueous-based polychloroprene adhesive, the increase reaches 122%.

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1. Introduction

It is hard to image any product at home, in the industry, transportation, or any other surrounding location that does not contain some kind of adhesive or sealant. Based on this fact, we can affirm that adhesives and sealants are present in all segments of the economy, and their performance is directly associated to the achievement of these sectors. Adhesives and sealants are remarkable materials, and technologies used for the production and application of these materials are equally extraordinary.

In the course of time, we find evidence of the use of adhesive-like substances such as production of animal glues, asphaltic material, tree resin, among others. The development of organic chemistry witnessed advances in new materials and methods, and the production of synthetic adhesives.[1–3]

At present, the choice of adhesives to promote binding of substrate, contrary to automated (mechanical) methods of binding, incorporates some advantages for the use of these

substances such as uniting multi-components of various widths, vibration absorption, mechanical resistance and joint capacity, minimizing material fatigue problems, reducing the total weight of the compound and having an accessible cost.[4]

The use of adhesives still offers some challenges such as the necessity to prepare the substrate, limited temperature of service, lesser strengths, and tenacity when compared with metallic joints, the fact that it does not allow dismantling and low resistance to pulling off strengths.[4]

Modern adhesives and sealants are generally formulated based on a polymer matrix, energy, pigments, stabilizers, plasticizers, and other additives necessary to confer specific characteristics to the final product. The process used may involve simple or sophisticated compounds, and its process is called copolymerization.[5]

With the availability of so many options of materials, it is necessary to list some factors to be considered when choosing a determined type of adhesive:

- The adhesive must be able to 'wet' the substrate;
- If the surface is of difficult moistening, it should be prepared in order to receive the adhesive;
- Adhesives must be more rigid than the substrate in order to avoid concentration of physical stresses on the adhesives;
- Following drying and hardening, the strength of adhesion must withstand environmental conditions;
- Both the adhesive and the application method must be of low cost.[5]

Other factors to be pointed out in the case of formulation and choice of adhesives are the capacity of adhesion and cohesion of those adhesives. The polymeric matrixes and some additives must promote or enhance these adhesive characteristics: adhesion means the resulting strength of the chemical bond between adhesives and substrate, while cohesion means the resulting strength that develops between molecules of the adhesive.[6,7]

The adhesive developed in this work denominated polychloroprene contact adhesive that is available in the market in two versions: solvent-based adhesives and aqueous-based adhesives. As the adhesive industry evolves, searching for high efficiency substances in accordance with the environmental demands, studies regarding substitution of organic solvents are intensified and today 58% of the adhesives produced make use of water as solvent.[8]

An analysis of this scenario shows that the polychloroprene contact adhesive is still not very well accepted by consumers because although both versions of this product are available, it is still favored in the solvent version, mainly due to its properties of open time and adhesion capability.

Based on this fact, a study was made bearing in mind the purpose of innovation of the formulation concept of the contact adhesive, using raw materials obtained from advanced technologies, along with the use of magnetic conditioning of the formulated adhesive aiming at improvement of the mentioned properties.[9]

1.1. Polychloroprene contact adhesive

Contact adhesives are derivatives obtained from polychloroprene, phenolic resins, resins derived from pitch with open time proportional to the bonding rate, having a very good initial adhesion, easy application, low cost, and good resistance to aging effect on bonds

and chemical degradation. They can be used in a large range of substrates, form a bond within themselves by application of light pressure and high cohesion efficiency. They are flexible with a solid content of 12–28% for solvent based and between 40 and 55% for aqueous based. They are used in furniture manufacturing and fabric-based components. These adhesives must be applied to both surfaces and allowed some time to dry before the two surfaces are pushed together.

Traditionally, contact adhesives require some time, called ‘open time’, to dry before the surfaces are to be held together. This open time can be defined as a maximum time interval for an adhesive film to remain dry, in contact with the air, and still maintain its auto adherent capacity (*tack*), under determined conditions of temperature and humidity.

In traditional formulations, both solvent based and aqueous based must respect this timing for evaporation of the solvent and the intensification of the cohesion forces. In the case of the organic solvent, timing is highly reduced and attributed to this type of adhesive is the characteristic of ‘immediate bonding’ whilst for the case of the aqueous-based adhesive timing varies from 45–60 min, thus generating idle time in the case of a production process.

Another characteristic of the adhesive that deserves attention is the adhesion capacity. Although commercial formulations have high adhesion capacity, i.e. are able to resist medium shearing tensions in the order of 2 kgf/cm², this figure is inferior to that observed in essays with solvent-based polychloroprene adhesives which average reference value is 3.5 kgf/cm².

To resolve this problem, it is necessary to find innovating alternatives for the choice of raw materials used to prepare the adhesive as well as introducing steps to the adhesive production process.[9]

1.2. Aqueous-based polychloroprene contact adhesive

Both the solvent-based polychloroprene contact adhesive as well as the traditional aqueous based are composed of polymer matrixes, resins, antioxidants, and other additives to obtain ideal characteristics for its application. In the aqueous-based formulation, normally a polymeric matrix is an emulsion in water. In this formulation, it is the water available in the emulsion that must evaporates to proceed to bonding.

However, nowadays there are polymeric matrixes that can be used jointly with nano-additives, substances that, in this size range, can extend some of their properties due to the outcome of its size.

However, within the nanoscience knowledge when you work with substances in this order of range, strengths such as gravity, friction and combustion remain in the background, whereas relevant aspects of the electrostatic forces, present in nanotechnology, are the forces of *van der Waals* forces and the Brownian movement. As mentioned before, the size effect can interfere in some properties of the materials, such as, for example, magnetic properties of fine particles can have their properties altered into a super paramagnetic state. Another situation within the nanotechnology can be generated by the increase in the superficial area of the nanomaterials resulting in a significant increase in its reactivity. This characteristic is very relevant for a substance also used as a thickener in a formulation.

Zinc oxide has some important properties in formulations of the polychloroprene contact adhesive, such as, for example, acting as an antioxidant, ultra-violet rays protector, bactericide, but in this special case, together with other additives, it will operate as a thickener,

interfering in the viscosity of the adhesive formulated and helping in the bonding process as well as acting against the aging of the structural joint and discoloration of the bonded substrates. This thickener propriety becomes favorable due to the size effect of the zinc oxide when used in a nanometric scale.

Another relevant factor for the development of aqueous-based adhesives for this type of polymer matrix is the possibility of avoiding to excessively relying on the solvent-based adhesive since it can be used as a narcotic substance, acting on the nervous system and causing chemical dependence. Besides reducing the use of organic solvents in formulations of the polychloroprene adhesives, one can also substitute the phthalates used as plasticizers and the catalysts with heavy metals.

The proposed use of the magnetic conditioning as one of the processes phase following the formulation of the aqueous-based adhesive relies on the fact that there are studies showing evidence such as pH modifications, electric conductivity, superficial tension, magnetic susceptibility, electrolytic dissociation, spins inversion affecting hydrogen connections, and alteration of the salts and crystals structure of solutions submitted to this process; in other words, when speaking of an aqueous-based adhesive, it is possible to obtain some desirable modification of the adhesive properties when magnetically conditioned.

Studies also report the influence of the magnetic field influence on polymeric matrixes containing zinc oxide in their structure in a nanometric scale, with the object of organizing and improving the mechanical properties of these polymeric matrixes, and, in the case of this essay, with the knowledge that oxide is susceptible to magnetic conditioning, its function as thickener in the adhesive formula can probably be intensified as a consequence of the increase in interactions between the polymer molecules aroused by the zinc oxide in a nanometric scale, promoting a crosslinking.[9]

1.3. Production of polychloroprene aqueous compositions

To produce compositions of aqueous dispersions of aqueous-based polychloroprene, the following proportions are conveniently mixed (Patent US 7659338)[10]:

- Principal Dispersion of polychloroprene: 50–9, 95% in weight.
- Secondary Dispersion of polychloroprene: 0–79%, 95 in weight.
- Hydroxylamine: 0.05 to 2% in weight.
- Zinc oxide Dispersion: 0 to 10% in weight.
- Silicon dioxide Dispersion: 0 to 40% in weight.
- At least one antioxidant: 0 to 10% in weight.
- Substances and ancillary additives.

After the formulation, the polychloroprene adhesive can be applied by a mixture pulverizing process where formulation of the aqueous adhesive and a coagulant (catalyzer) are transported separately in a spray gun, and finally mixed in the pulverized jet. The mixture is processed in the air and following the contact between the adhesive and the coagulant, glue is deposited on the substrate and the curing process is started. As already mentioned, the insertion of additives is necessary such as, for example, aqueous dispersions or polychloroprene formulations in order to provide formulation stability during storage as a guarantee for its use, or for protection of the adhesives against aging or discoloration.

1.4. Magnetic conditioning of aqueous solutions

The magnetic conditioning aqueous solution is a simple feature in which the aqueous solution passes through a magnetic field changing some of their physicochemical properties. This magnetic conditioning used to prevent sedimentation and removal of accumulated sediment in the aqueous solution was first patented by Vermeiren, Belgium, in 1953, and then recognized as discoverer of the fact that magnetic fields affect the properties of water and aqueous solutions. Since then, several studies have reported changes in the properties and behavior of aqueous solutions when conditioned magnetically such as pH, surface tension, reversing the spins that affect the hydrogen bonds, magnetic susceptibility, and electrical conductivity.

Based on these studies, it was decided to apply the magnetic conditioning in adhesives aqueous based, as some changes, such as pH, which aids in the adhesive clotting process, increasing its cohesive force and the change in surface tension amending polarity of the adhesive, making it less polar relative to the substrate, thereby assisting the wetting process, could result in improvements in adhesion capacity.[11–13]

2. Materials and methods

The methodology of this work can be defined as a bibliographical and documental, experimental and comparative research, therefore four groups of samples were organized each containing 25 sets of substrates. The groups of samples were designated as follows:

- Group 1 (A1): Samples in which solvent-based polychloroprene contact adhesive was applied, a *Colabras trademark*, produced by the company Brascola, acquired readily formulated for use.
- Group 2 (A2): Samples in which aqueous-based polychloroprene contact adhesive was applied, a *Cascola trademark*, produced by the company Henkel, acquired readily formulated for use.
- Group 3 (A3): Samples in which water-based polychloroprene contact adhesive was applied, formulated in accordance with technical directions from the Development Department of the company Convestro.
- Group 4 (A4): Samples in which water-based polychloroprene contact adhesive was applied, formulated in accordance with technical directions from the Development Department of the company Convestro, however, before application, the already formulated adhesive was submitted to magnetic conditioning.

2.1. Raw materials

- (a) Principal polymer matrix (Dispercoll C[®] 84 - Covestro) of rapid crystallization, providing mechanical resistance to the adhesive, *Brookfield* viscosity, LVR model (sp 3, 10 rpm) of 100 mPa s, specific mass 1.12 g / cm³, solids content (oven 180 °C, 15 min) equal to 55% and a pH approximately 13 and approximately molecular weight 2.38×10^4 g / mol (Covestro)[14];
- (b) Secondary polymer matrix (Dispercoll C[®] VPLS 2372/1 - Convestro), with slow crystallization allowing adhesion of non-polar surfaces and increasing the pot life

of the adhesive, *Brookfield* viscosity, LVR model (sp 3, 10 rpm) of 100 mPa s, specific mass 1.12 g/cm³, solid content (oven 180 °C, 15 min) equal to 58% and a pH approximately 13 and approximately molecular weight 2.38×10^4 g/mol (Covestro)[15];

- (c) An anionic aqueous solution of silicone dioxide (Dispercoll S[®] 3030 - COVESTRO), that, together with zinc oxide will act as thickeners, increasing the viscosity of the adhesive, its mechanical resistance and thermal resistance[16];
- (d) A derivative substance from diphenylamine in aqueous solution (Rhenofit DDA[®] 50 - *Rhein Chemie Rheinau GmbH*), used as antioxidant, avoiding stress cracking;
- (e) Zinc oxide dispersion in water, in the nanometric scale (VP Disp ZnO[®] 20 DW-*EVONIK Industries*), which average diameter should not exceed 150 nm.

2.2. Experimental procedure

Solvent-based and water-based commercial adhesives were applied to the test specimen comprising two substrates, one made of ipê (Brazilian wood) and the other of Formica laminate. Before applying the adhesives, these substrates were duly sandpapered and cleaned, as surface preparation procedure. After applying the commercial adhesives using a brush, and allowing for the necessary open time for each type of adhesive, bonding was made applying pressure, and after 72 h, the test specimen were submitted to traction experiment in order to check the adhesion capacity of the market adhesives. This phase was performed with the intention of obtaining data for future comparison with the adhesive formulated with the new nano-additive and magnetic conditioning proposal.

For formulation of the nano-additive adhesive, after weighing the raw materials, we proceeded with the mixture in a high torque mechanical agitator microprocessor, in accordance with the proposal presented in Table 1:

Following the formulation and waiting time of the prepared adhesive, a portion of the adhesive for application was separated without submitting it to magnetic conditioning, and the other portion was submitted to the magnetic conditioning process.

This magnetic conditioning was performed by a system called magnetic cell shown in Figure 1, comprising of containers for circulation of the adhesive, two submerged centrifugal pumps with an outflow of 340 liters ℓ/h, output of 7 W and 0.89 mca nanometry charge, hoses for transfer of the adhesive, a set of magnets iron-boron-neodymium which intensity of the magnetic field system presented a value of 2120 gauss, gaussmeter, and agitators. The process occurred during 3 h, time suggested in studies on magnetic conditioning of aqueous solutions. After this elapsed time, we were able to proceed with the application of the adhesive on the test specimens.

For both the magnetically conditioned as well as the non-magnetically conditioned adhesive application, we used specific equipment for bi-component adhesives, i.e. a special

Table 1. Formulation of aqueous-based polychloroprene contact adhesive.

Raw Materials	Individual Quantity (%)
Dispercoll C 84	59.32
Dispercoll C VPLS 2372/1	25.42
Rhenofit DDA 50	1.7
VP Disp ZnO 20 DW	0.85
Dispercoll S 3030	12.71

Source: DispercollC/Baypren Contact Adhesives, 2014.



Figure 1. Magnetic cell.
Source: Author's equipment.



Figure 2. Complete application system of the aqueous-based adhesive, catalyzed by CO₂.
Source: Author's equipment.

applicator gun which promotes a catalysis using carbon dioxide concurrently with the process of spray application.

The carbon dioxide catalyst was used to check the ideal pH for intensification of the coagulation and consequent formation of cohesion forces of the adhesive which will confer the mechanical properties and capacity of the adhesion.

The equipment used for aqueous-based polychloroprene adhesive application is shown on Figure 2.

3. Results and discussions

3.1. Viscosity

We can primarily observe that initial viscosities of the commercial aqueous-based adhesives as well as those of the formulation suggested by Covestro, without magnetic conditioning, were 350 mPa s. After the magnetic conditioning, the viscosity of the formulated adhesive

reaches 1100 mPa s; therefore, there has been a significant thickening of the aqueous-based adhesive, bearing a viscosity closer to the commercial solvent-based adhesive which was 2000 mPa s.

The magnetic conditioning aqueous solution, as literature, organizes the molecular structures, facilitating the performance of thickening substances that promote the approach of polymer molecules. With the higher viscosity, the adhesive efficiently wets the substrate, and the film formed does not penetrate too much in it, thus improving the conditions for adhesion. The raw material used in the formulation as a thickener and thus to increase the viscosity of the adhesive was zinc oxide. It has its magnetic condition changed to super paramagnetic when magnetically conditioning, and probably their reactivity in the set of substances in the formulation, has been increased. The crosslink between molecules of the polymer matrix, caused by the insertion of zinc oxide results in increased viscosity of the formulated adhesive, thereby increasing the adhesion ability.

3.2. Open time

A significant reduction of the open time adhesive can be observed. In the case of the commercial aqueous-based adhesive, we checked that after application of the adhesive, a waiting time of 45 min was required in order to bond the substrates, whereas in the case of the formulated adhesive, catalyzed with CO₂ and nano-additive, as soon as the adhesive was applied, the substrates were in a condition to be bonded. The open time of this adhesive is 5 min at the most.

The theory that underlies this modification of the open time is related to the presence of zinc oxide and Dispercoll S 3030 in nanoscale, because materials in this particle size range can increase secondary connections, increasing the strength of these bonds, for instance, *van der Waals* forces, mainly from polymeric matrices.

The presence of both, the zinc oxide as the Dispercoll S 3030, favored approach of molecules of the polymeric matrix, allowing for crosslinking between them, which results the elimination of water molecules to the substrate and therefore not necessary to wait for the evaporating the solvent to bond the substrates.

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In the case of the formulated adhesives, catalyzed with CO₂ and nano-additives, the open time is equivalent to the solvent-based adhesive which is still mostly used in the furniture industry.

3.3. Shear tension

With regards adhesion, the shearing tension results of the experiment are presented in Table 2:

The results of the traction test to check the shear tension supported by the test specimen are presented in Table 2.

Table 2. Results of Traction Tests – Shear Tension in kgf/cm².

Test Specimen	Group A1	Group A2	Group A3	Group A4
	Solvent-based commercial	Aqueous-based commercial	Aqueous-based formulated SCM	Aqueous-based formulated CCM
1	3.04	2.09	1.39	2.62
2	5.30	3.75	1.16	4.77
3	2.97	2.05	0.79	2.00
4	4.52	2.58	1.21	2.44
5	2.65	2.24	0.83	2.69
6	3.47	3.36	0.43	2.07
7	4.62	2.55	0.47	3.56
8	5.21	4.06	1.32	3.43
9	4.33	4.06	1.62	3.00
10	3.29	3.40	1.67	3.27
11	3.12	2.14	0.97	2.79
12	4.83	2.66	0.54	4.07
13	3.36	3.18	1.73	2.81
14	2.96	2.35	0.80	5.42
15	3.52	3.00	1.19	3.04
16	4.00	3.18	1.02	5.40
17	3.03	3.79	0.97	3.18
18	2.85	4.36	0.83	2.24
19	4.99	1.99	0.15	3.45
20	3.06	2.15	1.25	2.95
21	3.68	2.91	2.51	4.34
22	2.87	1.29	2.74	3.13
23	4.02	1.83	0.76	5.50
24	5.35	1.74	1.65	2.04
25	3.93	1.99	0.64	3.32
Average	3.80	2.75	1.15	3.34

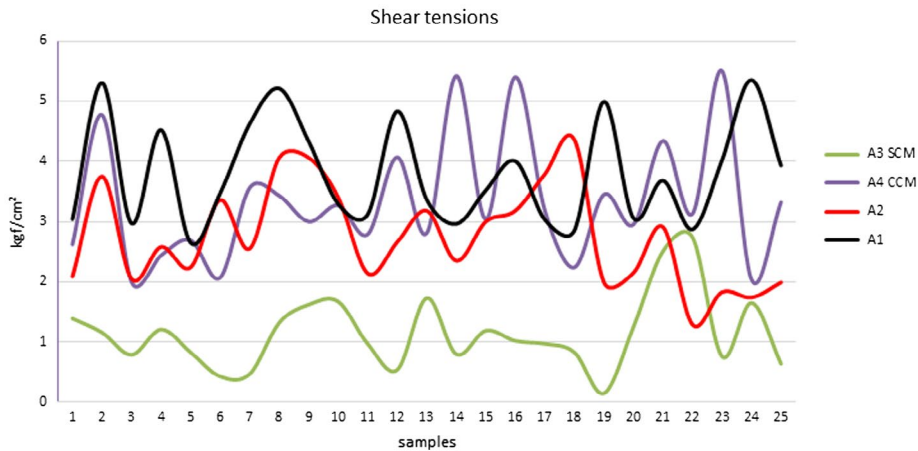


Figure 3. Results of the traction test for Groups A1 (Solvent base Adhesive Colabrás), A2 (Aqueous based Adhesive Henkel), A3 (Covestro SCM) and A4 (Covestro CCM).

Figure 3 presents a better visualization of the behavior of the shear tensions supported by the test specimen of the four groups of samples:

We note that the behavior of shear tensions of the formulated adhesive without magnetic conditioning remains below the results obtained from the other groups of samples. We were able to increase the capacity of adhesion of the aqueous-based polychloroprene adhesive to values very close to those reached by solvent-based adhesives.

Increased adhesion capacity can be applied as follows: after magnetic conditioning, there was an increase in the viscosity of the adhesive, a relevant factor in adhesion capacity. This resulted in a thicker film, favoring the wettability of the substrates, increasing the adhesion and cohesion strengths of the adhesive.

Therefore, the new contact adhesive formulation concept where it is not necessary to await solvent evaporation to promote substrates bonding using nano-additives and magnetic conditioning, applies adequate properties for this type of adhesive, making it competitive.

4. Conclusions

Based on this experiment, it can be checked that addition of the polymeric matrix using an antioxidant and thickener in the nanometer scale, together with a concomitant CO₂ catalyst system, reduced the open time of the water-based polychloroprene adhesive from 45 to 5 min, eliminating the problem of lack of efficiency in production. The theory behind this open time modification is related to the presence of zinc oxide in nanometric scale; as already described, materials in the granulometry scale can react on the secondary binding increasing the intensity of such bindings, especially the van der Waals between the polymeric matrixes, enhancing them, and thus do not need such an integrated solvent evaporation period similar to the one necessary in the commercial aqueous-based adhesive application.

The use of carbon dioxide as a catalyzer is very interesting since as we are dealing with an aqueous-based adhesive with insertion of CO₂, there is a carbonic acid process forming reducing the pH of the formulation and establishing the ideal adhesion situation.

However, we noted that this formulation did not reach the adhesion capacity as efficiently as the others.

This formulation was only efficient after magnetic conditioning, generating significant alteration of viscosity which varied from 350–1100 mPa s, without the necessity to add any extra thickener.

As for the adhesion capacity, it was evident that it was possible to enhance this capacity with the conditioning of the aqueous-based polychloroprene adhesive, since comparing the results of traction tests of the four groups of samples, we were able to approximate the result of the adhesion capacity of a water-based adhesive to a solvent-based adhesive, which was not observed between the commercial aqueous-based and solvent-based adhesives. As for the aqueous-based adhesives, the one magnetically conditioned was the one which presented the best adhesion capacity.

If we only compare the adhesives of same formulation, magnetically conditioned and not conditioned, the increase in the adhesion capacity reached 292%, a result beyond expectation, without inserting any other raw material to improve the characteristic of the adhesive. The open time for these two groups of samples did not suffer significant variation, but presented very significant results when compared to the commercial aqueous based adhesive.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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