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Barrier and Mechanical Properties of Clay-Reinforced Polymeric Nanocomposites

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In this work, clay-based nanocomposites films were prepared by addition of clay-Na$^+$ natural montmorillonite in pectin and hydroxypropyl methylcellulose (HPMC) matrices. Mechanical (tensile strength, elastic modulus, and elongation) and barrier (Water Vapor Permeability (WVP), and Oxygen permeability (O$_2$P)) properties were investigated. From results, it was observed that the WVP and O$_2$P decreased when nanoclay was included into the HPMC and pectin matrix films. Additionally, the incorporation of nanoclay in the films significantly improved the mechanical properties because the reinforcing effect of clay from its high aspect ratio and its enormous surface area. These results are very important in packaging area.

Keywords Barrier properties; Hydroxypropyl methylcellulose; Mechanical properties; Nanocomposite; Pectin

INTRODUCTION

Since the 1970s, environmental concerns have prompted several studies of different polar biopolymers as potential alternatives for synthetic polymers in the flexible packaging industries[1]. Numerous studies have been conducted investigating the properties of various protein, polysaccharide, and lipid-based biopolymer materials. Natural biopolymers have the advantage of being biodegradable, renewable, and often edible[2–3]. However, biopolymer films have exhibited relatively poor mechanical and water vapor barrier properties when compared to traditional polymeric films, therefore limiting their commercial use[4].

In the last years polymer/clay composites have received much attention, because of their extraordinary possibility to improve the barrier[5–8], and mechanical properties[9–12] of films. These composites are a class of hybrid materials composed of organic polymer matrices

and micro/nanoscale organophilic clay fillers[13] and due to their high aspect ratios and high surface area, if clay particles are properly dispersed in the polymer matrix at a loading level of 1–5% (w/v) unique combinations of physical and chemical properties will be obtained, that turn these composites attractive for making films and coatings for a variety of industrial applications[14].

Kumar et al.[15] study the improvement of tensile and flexural properties in epoxy/clay nanocomposites reinforced with weave glass fiber reel. The authors observed that mechanical properties were significantly increased due to an increase in clay content up to 5 wt%, and decreased with a further increase in clay content.

Polymer nanocomposites are also known as polymers that have been reinforced with small quantities of nano-sized particles (nanofillers). An important class of nanofillers involves nanoclays belonging to the smectite group, such as montmorillonite. Montmorillonite belongs to the family of 2:1 layered silicates. Its structure involves layers about one nanometer thick, each of which consists of two tetrahedral silica sheets fused to an edge-shared octahedral sheet of aluminium/magnesium oxide/hydroxide[16]. Among the clay minerals and clay-like materials used as filler for improving properties, the montmorillonite is the most common material because of its low cost and high availability, but also because montmorillonite presents a relatively high cationic exchange capacity and is easily expandable, which allows the intercalation of a wide range of organic species[17].

Cellulose derivatives are used in a wide variety of applications fields such as food, pharmaceutical, textile, and adhesive industries. Cellulose derivate such as hydroxypropyl methylcellulose (HPMC) is the most extensively employed because of its ease of use, low-coast, availability, water solubility, and non-toxicity[18]. They constitute one of the most dedicated polymers used in the production of packaging film[19].

Pectin is a natural, non-toxic and anionic polysaccharide extracted from cell walls of most plants. Pectin is the methylated ester of polygalacturonic acid. The pectin, by itself or by its gelling properties, was employed in pharmaceutical industry, health promotion and treatment. It has been used potentially as a carrier for drug delivery to the gastrointestinal tract, such as matrix tablets, gel beads, film-coated dose form[20].

The aim of the present study was to investigate the effect of addition of clay-Na+ natural montmorillonite on the mechanical, water vapor and oxygen permeability properties of pectin and HPMC films prepared by casting method. Different concentrations of clay were investigated in HPMC films to optimize the performance of the nanocomposites. A possible mechanism on the potential use of clay incorporated in edible polymers and their possible use in packaging has been proposed.

EXPERIMENTAL
Materials

High methoxyl pectin (degree of esterification 59–65%) is purchased from Systems BioIndustries, Fair Lawn, NJ. Hydroxypropyl methylcellulose (Methocel E15) was obtained from Dow Chemical Co. (Midland, MI, USA). Natural montmorillonite with an ion-exchange capacity of 92 mequiv/100 g (Cloisite Na+) was supplied from Southern Clay Products, Inc. All chemicals were used as received.

METHODS
Preparation of Natural Montmorillonite Suspension

The cloisite-Na+ was dissolved in distilled water (0.2% w/v) under magnetic stirring for 1 h. The suspension was centrifuged for 30 min at 5000 rpm and the supernatant was removing to film solution preparation. This step is important for purification of the solution.

Characterization of Cloisite-Na+

FT-IR Analysis

FT-IR spectrum of natural montmorillonite was taken with a Paragon 1000 Perkin Elmer Spectrum (Perkin Elmer Life and Analytical Sciences, Inc. Waltham, MA USA) in the range from 4000 to 400 cm−1. The FT-IR spectrum was used to characterize the clay properties. Powdered sample was prepared using KBr to form pellets.

X-Ray Analysis

The X-ray diffraction powder (XRD) pattern was collected using a Rigaku D/Max 2500PC X-ray diffractometer with a rotary anode using Cu Kα ($\lambda = 1.5406$ Å) radiation operating at 150 mA and 40 kV.

Nanocomposites Preparation by Casting

The pectin and HPMC films were obtained according to the procedure reported by Moura et al.[2] The polysaccharide solution (control film) was obtained dissolving 3.0 g of polysaccharide in 100 mL of distilled water under magnetic stirring for 12 h. A 3.0 (% w:v) polysaccharide solution was used in all film formulations. The films based on nanocomposites were obtained by addition of 3.0 g of polysaccharide in 100 mL of clay solution (both freshly synthesized), under magnetic stirring for 12 h. After the solutions were prepared, the flasks were allowed to rest for 6 h to degas in order to prevent microbubble formation within the films. The solutions were then poured on a glass plate (30 × 30 cm) covered with Mylar (Polyester film, DuPont, Hopewell, Va., U.S.A.) for film preparation by casting. The mixture were cast at a wet thickness of 0.5 mm onto plates using casting bars and the plates were placed on a leveled surface at room temperature and allowed to dry for 24 h. After drying, the films were removed from the Mylar and conditioned (for three days) in plastic bags at room conditions: 25 ± 1°C and 30 ± 2% RH.

Film Thickness

Film thickness was measured using a model 7326, digital micrometer (Mitutoyo Manufacturing, Tokio, Japan) at 5 random position of the film. The mean values were used to calculate water vapor and oxygen permeability and mechanical properties.

Tensile Tests

Films used for tensile tests were conditioned around at 30% RH and 24°C for 48 h before the measurements. These films (thickness of 0.03 mm) were then cut to have a rectangular dimension according to ASTM D882-97 (100 mm long and 15 mm wide)[21]: midsection 15-mm wide; 100-mm long, flaring to 25-mm by 35-mm square sections on each end. An Instron Universal Testing Machine (Model 1122, Instron Corp., Canton, Mass., U.S.A.) was used to determine the maximum TS (tensile strength), maximum elongation at break (%) and elastic modulus. The films were stretched using a speed of 50 mm/min. Testing conditions were 30 ± 2% RH and 24 ± 2°C. Tensile properties were calculated from the plot of stress (tensile force/initial cross-sectional area) versus strain (extension as a fraction of the original length)[22]. The mechanical properties were analyzed as a function of clay content and matrix-type.

Water Vapor Permeability (WVP)

WVP was determined by modification of the ASTM E96-92[23] gravimetric method to determine the relative humidity (RH) at the film according to the method used by McHugh et al.[24] Five films were cast from each treatment, in 8.0 cm internal diameter Teflon® plates. After

drying, one sample without defects was cut from each film. Distilled water (6 mL) was dispensed into flat-bottom Plexiglas® cups with wide rims. The film was sealed to the cup base with a ring using 4 screws symmetrically located around the cup circumference. The cups were placed in temperature-controlled cabinets at 25°C, containing fans and held at 0% RH using anhydrous calcium sulphate (W. A. Hammond Drierite Co., Xenia, OH, USA). Weights were measured periodically after steady state was achieved and used to calculate WVP according to the following equation.

Water vapor permeability (WVP) was calculated using the following relation:

$$WVP = \frac{WVTR}{(p_2 - p_3)} y \qquad (1)$$

where WVTR was obtained from the slope of the weight loss rate through the film surface and p_2 was the water vapor partial pressure of water vapor on the film underside; p_3 is water vapor partial pressure at the upper side of the film and y was the average film thickness. Water vapor permeability of each film was measured as the mean and standard deviations of 5 replications. Units for WVP were $cm^3 \ \mu m \ m^{-2} \ d^{-1} \ kPa^{-1}$.

Oxygen Permeability (O$_2$P) of Films

An Ox-Tran 2/20 ML modular system (Modern Controls Inc., Minneapolis, MN) was utilized to measure oxygen transmission rates through the films according to standard method D3985 (ASTM, 1995)[25]. Oxygen transmission rates were determined at 23°C and $55 \pm 1\%$ RH. Each film was placed on a stainless steel mask with an open testing area of $5 \ cm^2$. Masked films were placed into the test cell and exposed to 98% N_2 + 2% H_2 flow on one side and pure oxygen flow on the other. The system was programmed to have a 10 h waiting period to allow the films to achieve equilibrium. Oxygen permeability was calculated by dividing O$_2$ transmission rate by the difference in O$_2$ partial pressure between both sides of the film (1 atm) and multiplying by the average film thickness measured at 5 random places. Four replicates of each film were evaluated. Units for O$_2$P were $cm^3 \ \mu m \ m^{-2} \ d^{-1} \ kPa^{-1}$.

Statistical Analysis

Analysis of variance (ANOVA) was applied using Minitab 14.2 (Minitab Inc., State College, PA, USA) to determine significance of differences between means.

RESULTS AND DISCUSSION

The clay known as montmorillonite consists of platelets with an internal octahedral layer sandwiched between two silicate tetrahedral layers as illustrated in Figure 1[26].
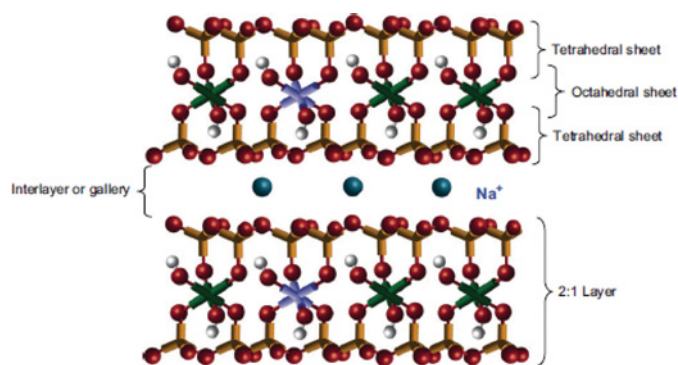

FIG. 1. Structure of sodium montmorillonite. Courtesy of Southern Clay Products, Inc. (Color figure available online.)

Figure 2 shows the XRD pattern of the Cloisite Na$^+$, while an intense diffraction peak at low 2 theta angles (7.80°) corresponding to a clay interlayer spacing value (d_{001}) of 1.13 nm (11.3 Å) can be visualized. FT-IR spectrum of Cloisite Na$^+$ is presented in Figure 3. The bands at $3630 \ cm^{-1}$ and at $3440 \ cm^{-1}$ were associated with the stretching modes of Si–OH and –OH groups of interlayer water; a band at $1642 \ cm^{-1}$ was attributed to the –OH bending mode of water; bands at $1042 \ cm^{-1}$ corresponding to the Si–O bending and stretching modes; bands at $526 \ cm^{-1}$ and $464 \ cm^{-1}$ corresponding to the stretching modes of Al–O and Mg–O, respectively[27].

A common reason for adding fillers to polymers is to increase the modulus or stiffness via reinforcement mechanisms described by theories for composites. Properly dispersed and aligned nanoclay platelets have proven to be very effective for increasing stiffness. The suitable use of nanocomposites is also strongly dependent on its favorable mechanical and barrier properties. Figure 4 shows the effect of clay content on tensile strength (TS) of HPMC
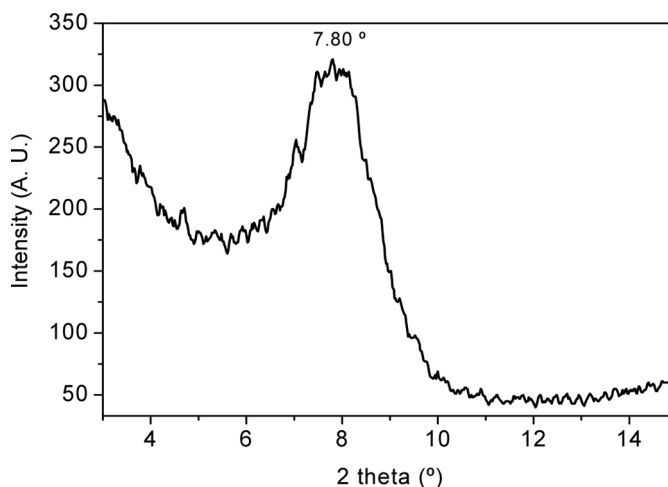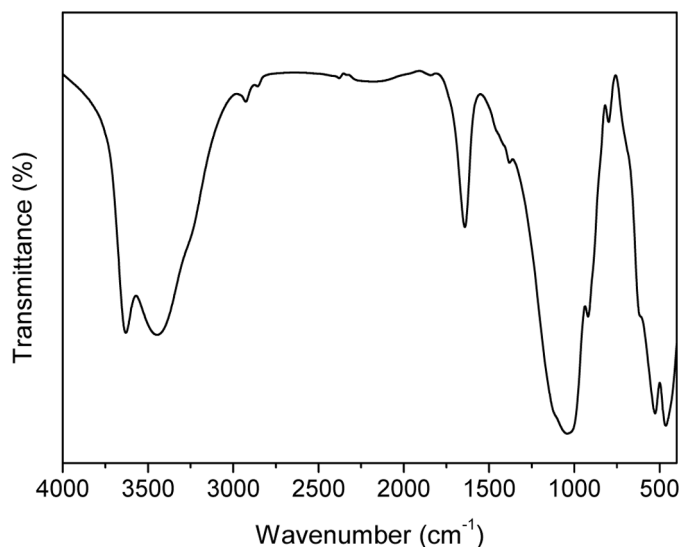

FIG. 2. XRD pattern of Cloisite Na$^+$.

FIG. 3. FT-IR spectrum of Cloisite Na+.

films. In HPMC films without clay, the tensile strength is 28.9 ± 0.9 MPa. When clay 2.5 (% w:v) were included in the HPMC films, the TS of the film was 55.2 ± 1.1 MPa.

For films containing clay 4.0 (% w:v), TS increased to 71.0 ± 1.3 MPa. This behavior is mainly due to reinforcing effect of clay from its high aspect ratio and its enormous surface area, which leads to high strength improvement. The other polysaccharide utilized was the pectin. In the pectin nanocomposite, the effect of addition of clay was the same observed in the HPMC films. In pectin films
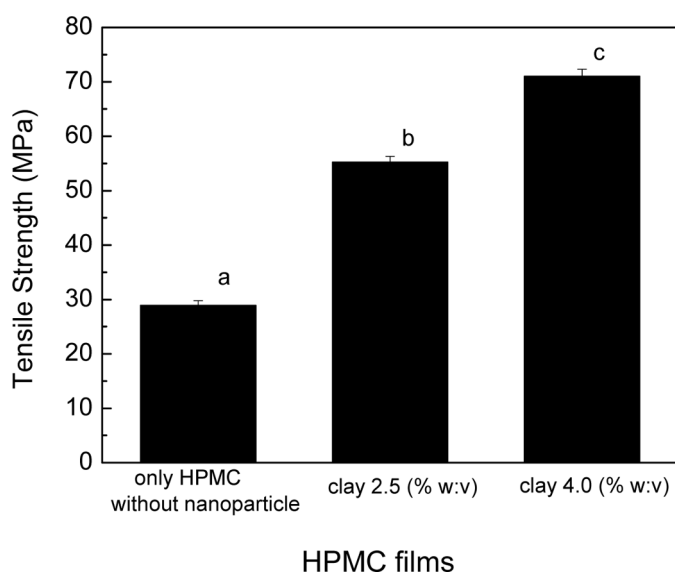

FIG. 4. Effect of clay content on tensile strength of HPMC films. Columns show the means and error bars indicate the standard deviations. Different letters within a column indicated significant difference at $p < 0.05$.

without clay, the tensile strength is 31.9 ± 0.8 MPa. When clay 2.5 (% w:v) were included in the pectin films, the TS of the film was 62.2 ± 1.0 MPa. Two-way ANOVA confirmed that the addition of nanoclay into HPMC films and its increase of concentration increased TS of the films. The clay fills in gaps in the films acting as reinforcing agents. In the literature, the same reinforcing effect has been observed in different matrices. For example, Sothornvit et al.[28] observed this effect in WPI/organoclay composite films.

The percentage elongation changed when clay specimen was added in films as shown in Table 1. The increase of the elongation improved the tenacity of the films. The elastic modulus increases with addition of the clay and present significant variation with different clay content. In addition, the elasticity of the films was preserved with addition of clay. In these ways, the addition of clay to HPMC and pectin films results in significant improvements in film mechanical properties. Probably, for the HPMC and pectin nanocomposites, the extent of the improvement of the modulus does not depend only upon the average aspect ratio of the dispersed clay particles. It seems from the results that the interaction between HPMC or pectin and clay is a relevant parameter for the stiffness improvement. Indeed the excellent modulus in the case of HPMC with 4.0% (w:v) of clay and pectin with 2.5% (w:v) of clay can be attributed to the strong interactions between polysaccharides matrix and silicate layers due to formation of hydrogen bonds.

Permeation is the mass transfer phenomenon that occurs when a molecule passes through a material or membrane from an area of high concentration to an area of low concentration. However, gas and solute permeation usually have their flux defined differently. Henry's law is applied to relate the surface concentration of a gas component with the partial pressure in the atmosphere in which the packaging material is in contact. Most permeable substances that affect the quality of food products are gases such as oxygen, carbon dioxide, noble gases, nitrogen and water

TABLE 1
Effect of concentration of clay on elastic modulus and % elongation of HPMC and pectin films

Type of film	Elastic modulus (MPa)	Elongation (%)
HPMC film	900 ± 34^{a}	8.1 ± 0.7^{a}
Pectin film	1884 ± 23^{a}	2.9 ± 0.1^{a}
2.5 clay HPMC	2461 ± 51^{b}	5.6 ± 1.4^{b}
4.0 clay HPMC	3643 ± 21^{c}	5.9 ± 1.2^{b}
2.5 clay Pectin	3844 ± 74^{b}	3.4 ± 0.1^{b}

*Different letters within a column indicated significant difference at $p < 0.05$.

TABLE 2
Oxygen permeability of HPMC and pectin films with
different clay content

Type of film	O_2 Permeability $cm^3 \, \mu m \, m^{-2} \, d^{-2} \, K \, Pa^{-1}$
HPMC film	182.4 ± 0.4^a
Pectin film	172.2 ± 0.3^a
2.5 clay HPMC	20.5 ± 0.5^b
4.0 clay HPMC	5.85 ± 0.3^c
2.5 clay Pectin	28.2 ± 0.2^d

vapor. These gases affect the rancidity, the ripening, and the hydration/dehydration of a food product, and generally determine the length of a product's shelf life. Therefore, the oxygen and water vapor rate transmissions are commonly used for quantifying the performance of packaging materials in industry[29].

Oxygen permeability (O_2P) of the HPMC and pectin films with and without nanoclay is summarized in Table 2. The O_2P of the control HPMC film was $182.4 \pm 0.4 \, cm^3 \, \mu m \, m^{-2} \, d^{-2} \, kPa^{-1}$. The O_2P decreased significantly when the clay was added in matrix film. Two-way ANOVA confirmed that addition of clay significantly ($P < 0.05$) decreased the oxygen permeability of the HPMC films. In the pectin films the same effect was observed whereas the O_2P of control film is $172.2 \pm 0.3 \, cm^3 \, \mu m \, m^{-2} \, d^{-2} \, kPa^{-1}$. Whenever, with addition of nanoclay (2.5% w:v) the new O_2P of the pectin film is $28.2 \pm 0.2 \, cm^3 \, \mu m \, m^{-2} \, d^{-2} \, kPa^{-1}$. Increasing the clay amount decrease O_2P through films because of the reduction free volume in the film network.

The WVP values, along with actual RH conditions at the undersides of films during testing, of the HPMC and pectin films with and without nanoclays are shown in Table 3. The WVP of nanocomposites films changed significantly ($P < 0.05$) depending on the concentration of nanoclays used. The decrease in WVP of polymer/clay

TABLE 3
Effect of presence of clay on WVP and % RH at
polysaccharide film underside

Type of film	WVP (g mm K^{-1} Pa^{-1} h^{-1} m^{-2})	RH at film underside (%)
HPMC film	0.794 ± 0.03^a	78.2 ± 0.7
Pectin film	0.415 ± 0.01^a	79.1 ± 0.3
2.5 clay HPMC	0.292 ± 0.01^b	79.0 ± 0.2
4.0 clay HPMC	0.207 ± 0.01^c	78.5 ± 0.2
2.5 clay Pectin	0.246 ± 0.02^b	79.2 ± 0.3

*Different letters within a column indicated significant difference at $p < 0.05$.

nanocomposites films is mainly attributed to the tortuous path for water vapor diffusion due to the impermeable clay layers distributed in polymer matrix that lead to an increase in effective diffusion path length[30,31]. The relative humidity at the film underside, indicated in Table3, had no significant difference ($79.2 \pm 0.6\%$ RH) for all the samples whereas the change in RH lead to a considerable changes in WVP results. The WVP of the control HPMC film was $0.794 \pm 0.04 \, cm^3 \, \mu m \, m^{-2} \, d^{-1} \, kPa^{-1}$.

The WVP decreased significantly when nanoclays were included in the HPMC matrix films. For example, WVP decreased to 0.292 ± 0.01 and $0.207 \pm 0.01 \, cm^3 \, \mu m \, m^{-2} \, d^{-1} \, kPa^{-1}$ for HPMC films with 2.5 and 4.0 (% w:v) nanoclay, respectively. In pectin matrix films of WVP value is $0.415 \pm 0.01 \, cm^3 \, \mu m \, m^{-2} \, d^{-1} \, kPa^{-1}$. After addition of nanoclay (2.5% w:v) in pectin matrix film the WVP value is $0.246 \pm 0.02 \, cm^3 \, \mu m \, m^{-2} \, d^{-1} \, kPa^{-1}$. Two-way ANOVA showed that the presence of nanoclay in the films decreased the WVP of the HPMC and pectin films. Important information is that an increase on the concentration of nanoclay, in HPMC films decreased the WVP values. In both matrixes the permeation of water molecules through these films is more difficult which results in a decrease in the WVP values.

CONCLUSIONS

The present study examined the incorporation of fillers of nanoclay into HPMC and pectin films. The incorporation of nanoclay in the films significantly improved the mechanical and barrier properties. The WVP and O_2P decreased when nanoclay was included into the HPMC and pectin matrix films due to increase of tortuosity pathway of solutes through the film matrix. Thus, the addition of clay to polysaccharide films is a promising way to prepare stronger and more stable films. In addition, the tensile, water vapor and oxygen vapor barrier properties of HPMC-based nanocomposites films varied depending of concentration of nanoclay utilized. The increased barrier and mechanical property suggests a great potential of the HPMC/clay and pectin/clay nanocomposites films in the application in food and beverage packaging.

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   www.tandfonline.com (the "Site") is owned and operated by Informa UK Limited ("Informa," "We," or "Us") whose registered office is at Mortimer House, 37-41 Mortimer Street, London, W1T 3JH. We are registered in England and Wales under company number 1072954. More…

Part II - Intellectual property and access and license types

10. **Becoming a registered user and use of passwords**

    Access to certain areas of the Site will be restricted to registered users only. You can apply to become a registered user here. More…

Part III - The public areas

14. **Use of public areas and content standards**

    From time to time, we may operate interactive areas on the Site in the form of blogs, wikis, comment forums, or message boards, where users can review topical debate and, sometimes, post comments and upload materials to the Site (the "Public Areas"). More…

Part IV - E-commerce

19. **Terms and conditions of supply**

    The following terms shall apply to e-commerce transactions taking place on or via the Site. More…

Part V - General provisions

26. **Written communications**

    Applicable laws require that some of the information or communications we send to you should be in writing. When using our site, you accept that communication with us will be mainly electronic. More…

For a downloadable copy, you can select the PDF here….