In-situ Analysis of Chemical Structure of API Adhesive Using FT-NIR Spectroscopy

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Abstract

Aqueous vinyl polymer solution-isocyanate (API) adhesive was prepared using polyvinyl alcohol (PVA), styrene-butadiene rubber (SBR) latex and polymethylene polyphenyl polyisocyanate (pMDI). The primary chemical reactions of NCO in API adhesive layer were detected by using Fourier transform near infrared spectroscopy (FT-NIR) and Fourier transform infrared spectroscopy (FT-IR). In addition, 2D correlation spectroscopy and 2nd derivatization of FT-NIR spectrum were also used. The consumption of isocyanate (2273 cm\(^{-1}\)), generation of urea (5005 cm\(^{-1}\)) and urethane (5065 cm\(^{-1}\)) linkage in post-cure process were conformed. The effect of storage humidity, PVA’s feature and SBR’s addition amount on chemical structure were also studied. The result showed that increase in degree of storage humidity or SBR’s addition amount resulted in an accelerating effect on NCO’s chemical reactions. The effect of PVA’s feature was also observed.

Key words : API adhesive/Urea/Urethane/FT-NIR/2nd derivative spectra

1. Introduction

Aqueous vinyl polymer solution-isocyanate (API) adhesive is environment friendly adhesive with no formaldehyde release and has been used widely for plywood, glulam, etc. in Japan. API adhesive was developed in Japan by Kuraray (Japan), Koyo (Japan) and Asahi Plywood (Japan) in 1971\(^1\),\(^2\). NCO in API adhesive has high reactivity so could react with active hydrogen at ambient temperature and establish cross-linking structure, by which high bond strength can be obtained. On the other hand, the pH of API adhesive is around 7, therefore, the erosion effect of API adhesive to wood is insignificant. However, the high cost and short pot life may limit the application of API adhesive.

The components of API adhesive are water-based emulsion, isocyanate cross-linker and filler. Water-based emulsions are generally including polyvinyl alcohol (PVA) aqueous solution, styrene-butadiene rubber (SBR) latex or ethylene vinyl acetate (EVAc) emulsion\(^3\), etc. Isocyanate cross-linker is usually toluene diisocyanate (TDI), 3-isocyanatomethyl-3, 5, 5-trimethyl cyclohexyl isocyanate (IPDI) or polymethylene polyphenyl polyisocyanate (pMDI)\(^1\),\(^6\). Fillers, which can reduce the cost of the API adhesive and improve gap filling property, heat resistance property, are generally wood fiber, shell flour, clay and calcium carbonate (CaCO\(_3\))\(^5\). NCO in API adhesive could react with any active hydrogen, thus, the chemical structure of completely cured API adhesive is very complicated. But the primary cross-linking reactions are between NCO and PVA’s hydroxyl group or water, which generate urethane linkage and urea linkage, respectively. In this research the variation of API adhesive’s chemical structure attributed to NCO’s reactions was studied.

Both urethane and urea linkage generate from the reactions of hydroxyl groups, but the mobility of PVA and water are extremely different, therefore, the generation rate of urea linkage is much higher\(^6\). After API adhesive was applied to adherend, the water in
adhesive evaporates gradually. API adhesive turns from liquid-state into solid-state, and finally adhesive layer forms. This period usually takes one day and generally is called cure of the adhesive. Actually, cross-linking reactions of NCO could continue for many days even though the adhesive is in solid-state, and this period is called post-cure. In the post-cure period, adhesive layer absorbs moisture from ambient air then provides water for the urea generating reaction, in this term urethane generating reaction also continues. The post-cure process is very important for API adhesive because most of the chemical linkages, especially urethane, are establishes and the adhesive layer’s mechanical properties improve in this period. But until now there still no definitive conclusion reveals how many days the post-cure reaction could last\(^5\).

Fourier transform infrared (FT-IR) spectrometer is normally used to analyze the chemical structure. Basing on FT-IR spectra, the generation of urea and urethane linkages were confirmed by Patel and Gao\(^7,8\). However, in FT-IR measuring process, it is difficult to repeat analysis of the same sample. On the other hand, Fourier transform near infrared (FT-NIR) spectrometer has the advantages such as simple pretreatment, damage treatment less and in-situ analysis compared to FT-IR, which detects the overtone and combination tone of bond’s vibration\(^9,10\). Therefore, FT-NIR was used to analyze the in-situ chemical structure of API adhesive layer in this study.

### 2. 2\(^{\text{nd}}\) derivatization

Derivatization is normally utilized as one effective analysis method for IR spectrum. By using 1\(^{\text{st}}\) derivatization the resolution is enhanced and the change of gradient in IR spectrum is calculated. 2\(^{\text{nd}}\) derivative spectrum is achieved by performing another derivatization on 1\(^{\text{st}}\) derivative spectrum, in which the negative peak shows up at the same position as raw IR spectrum. Especially for NIR spectrum, in which bands overlapping occur frequently, 2\(^{\text{nd}}\) derivatization could be used to separate the overlapping bands and eliminate the baseline draft effect.

### 3. 2D correlation spectroscopy

Two-dimensional (2D) correlation spectroscopy for IR is one analytical technique to study the molecular interactions basing on time-resolved IR spectra and started by Noda\(^11\). The 2D correlation concept comes from 2D NMR, however, 2D correlation spectroscopy for IR is different from NMR’s. 2D NMR is calculated from spectra with multiple pulse excitations, but in IR detection this procedure is difficult to be accomplished. In 2D correlation spectroscopy for IR the excitations are various temperature, time etc., which are easy to be collected. In this study the 2D correlation spectra were calculated by using 2D shige\(^13\), synchronous and asynchronous spectrum are obtained separately. In synchronous spectrum the auto-peak appears on diagonal, by which the change of band in IR spectrum corresponding to excitation is observed. Cross-peak generates from two correlative bands, therefore, basing on the signs (positive or negative) of cross-peaks in synchronous spectra and asynchronous spectra two bands’ changing rates can be confirmed. As for FT-NIR spectrum the 2D correlation spectroscopy is inappropriate to be performed directly on raw spectrum in which bands of different chemical linkages overlap significantly. In this study 2\(^{\text{nd}}\) derivatization was used to separate the overlapping bands in FT-NIR spectrum, further, 2D correlation spectroscopy was performed on 2\(^{\text{nd}}\) derivative spectrum.

### 4. Experimental

Materials: PVAs used in this experiment were purchased from WAKO (Japan) (PVA’ degree of saponification and MW were shown in Table 1). SBR latex was used as softener, supplied by NIPPON A & L (Japan) and has solid content of 51.7\%, styrene/butadiene molar ratio of 3/7. Cross-linker MR-200 (pMDI) was supplied by Nippon Polyurethane (Japan) (Scheme 1). Filter paper (Whatman) used was 100\% cellulose composite. CaF\(_2\) board (δ : 1mm, Φ: 25mm) used for FT-NIR spectrometer was purchased from Pier Optics (Gumna, Japan).

Preparation of API adhesive: At first PVA was dissolved in water to prepare 15\% PVA aqueous solution.
The PVA aqueous solutions’ viscosity were measured using HAAKE Rheostress 600 (Fig. 1). PVA aqueous solution and SBR latex were mixed together, stirred for 1 min and degassed for 30 s. Then pMDI was added to the PVA-SBR mixture, stirred for 2.5 min and degassed for 30 s. Mixing process was performed by using Hybrid Mixer HM-500 and the weight ratio of PVA: SBR: pMDI was 2:2:1.

Preparation of samples: For FT-NIR spectrometer CaF₂ board sample, Paper sample and film sample were prepared. 1. CaF₂ board sample: roll tape around the CaF₂ board to make a CaF₂ board container like a small cylinder, then the API adhesive was cast onto it. One day later peel the tape off (the adhesive film still sticks onto the CaF₂ board, the thickness of adhesive was about 0.4 mm, Fig. 2 a); 2. Paper sample: bond two pieces of filter paper together with API adhesive, the filter paper was cut into the size of 1×1 cm², the API adhesive’s amount for each paper sample was about 0.013 g (Fig. 2 b); 3. Film sample: cast API adhesive onto teflon sheet, one day then the adhesive film was peeled off (film’s thickness was about 0.23 mm, Fig. 2 c). For FT-IR spectrometer a very thin API adhesive film was prepared by casting API adhesive onto teflon sheet and then peeling the thin adhesive film off after one day drying (film’s thickness was about 0.03 mm, Fig. 2 d).

Storage humidity: each sample above is prepared in triplet and kept in three humidity conditions respectively: 1) normal (23 °C, 50 %); 2) dry (23 °C, -0 %), desiccator with phosphorous pentoxide; 3) moist (23 °C, -98 %), desiccator with potassium sulphate’s saturated aqueous solution.

Measurement: FT-NIR spectrometer NICOLET-6700, beam splitter CaF₂, resolution 8 cm⁻¹, 512 scans, 4000-12000 cm⁻¹. FT-IR spectrometer NICOLET-6700, beam splitter KBr, resolution 4 cm⁻¹, 128 scans, 400-4000 cm⁻¹. The background for paper sample was overlapped two pieces of filter paper, for film sample blank background, and for CaF₂ board sample one piece of CaF₂ board.

5. Results and discussions
5.1 FT-IR, thin API adhesive film
The chemical reactions of NCO take place right after the API adhesive was prepared and could last for several days, which are listed in Scheme 2. Actually at low temperature (< 60 °C), the formation reactions of biuret and allophanate linkages are very limited, so
the products at ambient temperature are almost only urea and urethane linkages. Fig. 3 shows the FT-IR spectra of thin API adhesive film of 3rd, 5th, 11th day. In Fig. 3 the band at 3334 cm\(^{-1}\) is attributed to N-H stretching vibration, the band at 3000-3150 cm\(^{-1}\) C-H stretching vibrations of alkene and benzene ring, the band at 2800-3000 cm\(^{-1}\) C-H stretching vibrations of methylene and methyl, respectively. NCO’s reactions continued for several days in storage period and this phenomena was conformed in Fig. 3, urea (1639 cm\(^{-1}\)) and urethane’s (1715 cm\(^{-1}\)) peaks’ intensity increasing with time, on the other hand, NCO’s peak decreasing. The consumption of hydroxyl group of PVA was monitored by detecting the peak at 1108 cm\(^{-1}\) of which peak’s intensity showed decrease with time. N-H’s peak at around 3334 cm\(^{-1}\) includes both urea and urethane’s information and showed an increasing tendency. Methyl, methylene, alkene and benzene ring do not react in API adhesive, so those peaks were considered to be totally changeless in the whole storage period. The peaks at 3000-3150 cm\(^{-1}\) corresponding to alkene and benzene ring show constant absorbance. However, the peaks of Methyl and methylene at 2800-3000 cm\(^{-1}\) are found to have a little variation, which could be attributed to the baseline drift of spectra.

Fig. 4 shows the synchronous and asynchronous 2D correlation spectra generated from FT-IR spectra. The
region was narrowed to 1000-3500 cm\(^{-1}\), in which the information of NCO, hydroxyl group of PVA, urea, urethane and N-H were presented, and the resolution was improved. Further, to clarify the sign (positive or negative) of cross-peak, the positive peak was marked by triangle and negative peak was marked by circle.

In Fig. 4a (synchronous spectra), auto-peaks of NCO, urea, urethane appear at 2274 cm\(^{-1}\), 1639 cm\(^{-1}\), 1715 cm\(^{-1}\) separately on diagonal (shown by arrows), indicating that these bands’ intensity were varying significantly. N-H and hydroxyl group’s auto-peaks are comparatively weak and barely show up. The negative cross-peaks of NCO-urea (2274-1639 cm\(^{-1}\)) and NCO-urethane (2274-1715 cm\(^{-1}\)) implies that urea and urethane’s band intensity changed in the opposite direction to NCO’s (NCO’s band intensity decreased while urea and urethane’s, on the other hand, increased with time as chemical reactions processed). In addition, the two cross-peaks of NCO-O-H (2274-1108 cm\(^{-1}\), positive) and NCO-N-H (2274-3334 cm\(^{-1}\), negative) are detected, verifying the variation of O-H and N-H’s bands (The positive cross-peak of NCO-OH suggests that hydroxyl group’s band intensity varied in the same direction as NCO’s; the negative cross-peak of NCO-N-H suggests that N-H’s band intensity varied in the opposite direction to NCO’s).

Moreover, the same signs (both positive or both negative) of cross-peaks in synchronous spectra and asynchronous spectra implies that the high wavenumber bond’s band intensity varies faster; the opposite signs (one positive and one negative) of cross-peaks in synchronous spectra and asynchronous spectra suggests that the low wavenumber bond’s band intensity varies faster. And this rule is just suitable for left area of diagonal in this paper. For example, basing on the negative cross-peak in Fig. 4a (synchronous spectra) and the positive cross-peak in Fig. 4b (asynchronous spectra) of NCO-urea (2274-1639 cm\(^{-1}\)), we could tell that urea’s (low wavenumber) band intensity varied faster. Further, the two negative cross-peaks in synchronous spectra and asynchronous spectra of NCO-N-H (2274-3334 cm\(^{-1}\)) denotes that N-H’s (high wavenumber) band changed faster than NCO’s.

Basing on FT-IR spectra the consumption of NCO and hydroxyl group of PVA, the generation of urea and urethane linkage were confirmed. But as we mentioned above the thin API adhesive film is not a real bonding system because no adherend is involved. Thus, FT-NIR spectroscopy was utilized in the further study.

5.2 FT-NIR, CaF\(_2\) board sample

By using FT-NIR spectrometer, the chemical structure of API adhesive layer in CaF\(_2\) board sample was in-situ detected. Fig. 5 is the FT-NIR spectra of CaF\(_2\) board sample in the region of 4000-12000 cm\(^{-1}\) for 1\(^{st}\), 8\(^{th}\), 11\(^{th}\) day. The band at 5665 cm\(^{-1}\) is attributed to C-H stretching vibration of SBR, the band at 4750-5100 cm\(^{-1}\) urea and urethane linkages\(^{10}\). Actually the bands of FT-NIR spectra are combination tone and overtone of bonds’ vibration, thus, the bands’ overlapping and baseline draft in FT-NIR spectra make the functional

![Fig. 5 The FT-NIR spectra of CaF\(_2\) board sample for 1\(^{st}\), 8\(^{th}\), 11\(^{th}\) day, (PVA II, normal humidity).](image-url)
groups’ assignment difficult. For these cases, 2nd derivatization is normally performed on FT-NIR spectra. 2nd derivative spectra is showed in Fig. 6, of which the useful bands appear at the region of 4500-6000 cm\(^{-1}\), SBR’s peak, urethane’s peak and urea’s peak are detected at 5665 cm\(^{-1}\), 5065 cm\(^{-1}\) and 5005 cm\(^{-1}\), respectively. By 2nd derivatization effect of baseline drift was eliminated, overlapping bands were separated, and two downward peaks of urea and urethane linkage appeared significantly. Moreover, urethane’s peaks and urea’s peaks show increasing tendency with time, which denotes that urethane and urea linkages were generating continuously in the storage period. Styrene-butadiene rubber’s peaks at 5665 cm\(^{-1}\) show changeless intensity values due to the reactionless property of SBR, which was considered that could be used as a normalization standard in the further study.

CaF\(_2\) board sample provides a perfectly even adhesive layer and is more suitable for FT-NIR analysis than the other two samples (paper sample and film sample), however, for most of API adhesives with a high viscosity it’s difficult to be prepared into a CaF\(_2\) board sample. Moreover, CaF\(_2\) board sample’s API adhesive layer just has one side contacting with storage environment, so the less moisture absorption was also considered. For film sample and paper sample, which do not have even thickness API adhesive layer as CaF\(_2\) board sample, the SBR’s peak at 5665 cm\(^{-1}\) was utilized as a standard to normalize 2nd derivative spectra.

5.3 FT-NIR, paper sample and film sample
The result of paper sample and film sample are shown in Fig. 7 and Fig. 8. In order to explain the effect of normalization, the raw 2nd derivative spectra without normalization is shown in Fig. 7 b (just SBR’s peaks), in which SBR’s peaks of different storage time disperse randomly attributing to uneven thickness of API adhesive layer in paper sample. Paper sample’s 2nd derivative spectra normalized by using SBR’s peak is shown in Fig. 7 a, SBR’s peaks overlaps perfectly, which shows a same effect as CaF\(_2\) board sample. In Fig. 7 a the increasing of urethane and urea peaks’ intensity with time is observed revealing that the crosslinking reactions were continuing with time. In addition, urea’s peak is more visible than urethane’s, and this can be explained as that urea generating reaction is faster. Actually NCO (isocyanate group) does react faster with water than with PVA and cellulose’s hydroxyl group, of which the result was proposed in previous study\(^8\). FT-NIR spectra and 2nd derivative spectra provided a new way to analyze the chemical structure of adhesive layer sandwiched between two pieces of filter paper, in other words, the in-situ analysis of API adhesive layer was accomplished. However, basing on FT-
NIR spectra it’s difficult to assign NCO and hydroxyl groups which are overlapping with other bands. In the region of 4540-4690 cm\(^{-1}\) three peaks’ decreases with time are observed and this phenomenon could be attributed to NCO's consumption, however, SBR also has strong absorbance in the region and it’s difficult to separate NCO’s absorbance from SBR’s. Paper sample was prepared to imitate the wood-bonding structure, film sample could reveal the chemical structure of pure adhesive layer. In this experiment the detection of reaction between NCO and hydroxyl group of filter paper’s cellulose was also expected, so the paper sample and film sample were utilized as a contrast group to investigate the reaction of cellulose. Fig. 8 is the result of film sample, where SBR’s peak (5665 cm\(^{-1}\)), urethane’s peak (5065 cm\(^{-1}\)) and urea’s peak (5005 cm\(^{-1}\)) are detected, and the significant difference of 2\(^{nd}\) derivative spectra between film sample and paper sample (Fig. 7 a) was not found. Further, this phenomenon proves that the in-situ analysis of adhesive layer in paper sample is feasible. However, 2\(^{nd}\) derivative spectra just show the rough change of the urea and urethane’s peak, and no more elaborate result could be summarized from it. For these case, the calculation of peak’ intensity was considered as a new method to investigate the chemical structure in detail.

5.4 FT-NIR, different humidity

API adhesive layer absorbs moisture from air to support cross-linking reaction, so the storage humidity has effect on the linkage forming reactions \(^{15-17}\). In this section three types of storage humidity (normal 50\%RH, dry -0 \%RH, moist -98\%RH) were utilized to discuss the effect of different humidity. To study the cross-linking reaction in detail, the intensity value of both urea and urethane’s peak in 2\(^{nd}\) derivative spectra were calculated, and the intensity value of urea vs. time was shown in Fig. 9. In initial 30 days, the moist’s urea is observed to generate faster than both normal’s and dry’s. Urea linkage generates from the reaction between pMDI and water, thus, this reaction is highly sensitive to humidity. In other words, in initial 30 days the moisture environment accelerated urea’s producing reaction in adhesive layer, on the contrary, dry environment limited the reaction. Moreover, the moist curve showed a steep slope in initial 30 days and a flat after this period, however, normal and dry curve do not have such a significant change in slope through the whole 60 days.

Urethane’s result is shown in Fig. 10, in which urethane does not generate as large amount as urea, therefore, these curves are not so notable. However the fastest generating rate of moist curve is also confirmed, also, dry curve shows the minimum amount. Urethane linkage generates from the reaction between PVA’s hydroxyl group and pMDI’s NCO and is not related to storage environment’s humidity directly. The result of Fig. 10 could be interpreted as that in the moist sample molecular chain is much more flexible, so it is easier for NCO to meet and react with both PVA’s hydroxyl group and water. Further, the large amount of urea linkage generating in moist sample could drag molecular chains closer, of course including PVA and pMDI, then provide them more chances to react.

Moisture can accelerate the linkage producing reaction but this acceleration is limited in a short period (in the experiment it is 30 days), and two points were proposed to explain the flat part of moist curve in Fig.
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Fig. 10: 1. the NCO was depleted until 30\textsuperscript{th} day so the cross-linking reaction totally stopped in the flat part; 2. the isocyanate group was not depleted, but the reaction became to be very slow in the flat part. To verify these two points the 2D correlation of 2\textsuperscript{nd} derivative spectra were utilized. Fig. 11 is the 2D correlation spectra calculated from 2\textsuperscript{nd} derivative spectra corresponding to the flat part of moist curve. On diagonal of Fig. 11 the auto-peak of neither urea nor urethane shows up (circle mark), which implies that in the flat part of moist curve both urea and urethane linkage barely generated, and the variation of peak’s intensity was hardly to be detected. NCO’s IR-absorbance appears at 2274 cm\textsuperscript{-1} \cite{18,19}, so the NIR-absorbance corresponding to overtone was consider to be detected at around 4550 cm\textsuperscript{-1}. At the lower right corner of Fig. 11, the small cross-peaks of urea and NCO appears (elliptical mark), which suggests that the reaction of NCO generating urea, even though very slowly, was still advancing in the period of 31\textsuperscript{st}-60\textsuperscript{th} day. And the difference in reaction rate between 1\textsuperscript{st}-30\textsuperscript{th} day and 31\textsuperscript{st}-60\textsuperscript{th} day was thought as relating to the change of molecular chain’s mobility caused by cross-linking \cite{14,20}.

5.5 FT-NIR, different PVAs

Most of PVA used for API adhesive is not completely saponified and the normal value of DS (degree of saponification) is 80-95\%. DS is one important index to the character of PVA, therefore, the effect of DS on urea and urethane generating reaction was studied in this section. To advance this study, three types of PVAs with different DS were used, of which the features were listed in Table 1 and Fig. 1, the DS increasing from 86\% to 98\% (the higher DS the more hydroxyl groups attaching to the PVA’s molecular chain). Urethane linkage generates from the reaction between NCO and PVA’s hydroxyl group, so the high DS PVA was assumed at first to produce more urethane linkages. Viscosity values of 15\% PVA aqueous solutions is shown in Fig. 1, which is related to molecular weight, molecular chain’s mobility as well as the reactivity of PVA, therefore, PVA aqueous solution with low viscosity was also expected to generate more urethane linkage in adhesive layer.

In Fig. 12, urethane’s data, the roman numerals from I to III present three different API adhesives prepared from three types of PVAs. As the result, three adhesives have different amount of urethane linkage, this indicates that the urethane generating rates are varying basing on PVA’s species. The maximum amount of urethane appeared in API II instead of API I, which goes against with our assumption, in other words, the highest DS PVA did not produce the maximum amount of urethane. One explanation was proposed that the low DS PVA have more hydrophobic group (acetyl group) attaching to molecular chain, so these PVAs could be more miscible with pMDI, and the chemical reaction of low DS PVA may carry on more smoothly. On the other hand, PVA II has a lower molecular weight and viscosity value, therefore, in API adhesive layer PVA II’s molecular chain is more flexible and has a higher reactivity. API II produced more urethane than API III in Fig. 12, and this phenomenon could also be attributed to the low viscosity value of PVA II aqueous solution. The result of this section suggests that the PVA with lower viscosity value has more flexible molecular chain, hence, the
hydroxyl group of PVA could have more chances to react with NCO.

5.6 FT-NIR, different SBR’s addition amount

For the preparation of API adhesive, three raw materials of PVA aqueous solution, pMDI and SBR latex were utilized. As interpreted above, PVA, pMDI and water participate in the cross-linking reactions, on the contrary, SBR, which was used as softener to improve the mechanical behavior of API adhesive, does not react in the whole storage period. But we suspected that SBR may have some influence on those cross-linking reactions. In liquid state SBR micelle has both hydrophilic group and hydrophobic groups on the surface so the interaction between SBR micelle and other components were also considered. In this section five types of API adhesives were prepared with various weight percent of SBR latex changing from 10% to 35.8%, and the weight ratio of PVA:pMDI was still 2:1. From 2nd derivative spectra of 1st-20th day, urea and urethane peak’s intensity were calculated as mentioned above. To normalize these spectra with different SBR content (SBR’s peak was used as standard), we further divided these peak’s intensities by PVA’s weight percent of each sample in this section. After all treatments these API adhesives with different weight percent of SBR were able to be compared and the results are shown in Fig. 13 and Fig. 14.

In Fig. 13 the maximum result appears in 35.8% and followed by 30.8%. It seems like the API adhesive with more SBR could produce more urea linkage in the storage period and the difference between five API adhesives became larger with time. Urethane’s result in Fig. 14 is similar to urea’s, and the maximum amount also arise in 35.8%. pMDI has only hydrophobic group (NCO) attaching to the molecular chain, on the other hand, PVA’s molecular chain is branched by a large amount of hydrophilic groups (hydroxyl group) and just a small amount of hydrophobic groups (acetyl group), therefore, the compatibility between pMDI and PVA is not very well. SBR latex is produced by emulsional polymerization and emulsifier is used in this process. In SBR latex emulsifier is still contained, attaching onto the surface of SBR micelle, thereby, SBR micelle has both hydrophobic group and hydroxyl group on the surface. For this reason SBR micelle mixed with PVA and pMDI could act as intermediary, and improve the compatibility of all components. In this case the well mixed NCO and hydroxyl group in the API adhesive with high SBR’s weight percent could react better in the storage period. In addition, SBR enlarges the volume of adhesive layer, the moisture absorbance increasing, and then the reaction of NCO and water processes more smoothly.

For most of commercial API adhesives SBR is mixed in the adhesive emulsion, not only the decreasing in cost but also some improvement in chemical structure aroused by SBR have been expected. Through this section we verified that the softener SBR could also act as an accelerator for NCO’s reactions.

6. Conclusion

In this study, FT-NIR was utilized to in-situ analyze the chemical structure of API adhesive layer in sandwiched paper, film and CaF$_2$ samples. Basing on the 2nd derivative spectra we confirmed that three factors
of humidity, PVA’s feature and SBR latex’s addition amount all have influences on the cross-linking reactions: Moist storage condition supplies more water for the adhesive layer than normal, dry storage condition then could accelerate cross-linking reactions; Urethane’s generating amount varied basing on PVA’s feature; And the addition of SBR latex can accelerate the cross-linking reactions.

References

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