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EFFECTS OF WOOD FLOUR AND CURING TEMPERATURE ON SOME PROPERTIES OF THE POLYURETHANE FOAM CORE IN A WOOD-BASED SANDWICH PANEL

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Abstract: The objectives of this study were to examine the chemical, physical, and mechanical properties of polyurethane (PU) foam prepared with various wood flour contents and curing temperatures for use as core layer in wood-based sandwich panels. The five alternative wood flour contents tested were 2, 4, 6, 8, 10, and 12% and the alternative curing temperatures 100, 120, 140, and 160°C were studied with 10% wood flour content. The results showed that the cell size and density of the PU foam increased at wood contents above 6%, which reduced the compressive strength of the samples. It was also found for PU with 10% wood content that curing at 100 to 160°C caused a decrease in foam density by 50%, compared with PU foam cured under the standard conditions (i.e., at room temperature). SEM and cell size measurements show the anisotropy of PU foam and the increase of foam cell diameter when increasing the curing temperature from standard condition to 140°C. Consequently, the compressive strength and its specific value declined abruptly at elevated curing temperatures, starting from 100°C. These results suggest that elevated curing temperatures cause degradation of the properties of woodfilled PU foam, due to effects on foam cell structure.

Key words: *polyurethane rigid foam; compressive strength; curing temperature; wood flour; sandwich panel.*

1. Introduction

Polyurethane (PU) is typically prepared by reacting the two basic components, isocyanate and polyol. For water-blown PU, the foaming process involves two competing reactions, namely the blowing and the polymerization reaction. The polymerization reaction of both components (i.e., isocyanate and polyol)

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forms urethane linkages (-RNHCOOR'-). In the blowing reaction, water reacts with isocyanate to form carbamic acid (R-NHCOOH) as a primary addition product. As this is unstable, it further sheds off amine and CO_2 molecules. The generated CO_2 gas is capable of filling pores, expanding the cellular structure and the PU matrix polymer. On the other hand, the amine immediately reacts with additional isocyanate to form urea, as shown in reaction (1). Further reactions of urethane and urea with additional isocyanate form allophanate and biuret, respectively. Due to the high reactivity of isocyanate, PU utilizes polyfunctional reactants, which leads to the formation of a cross-linked polymer network.

R-NCO	+	R'-OH →	R-NHCOO-R'				
Isocyanate		Polyol	Urethane				
R-NCO	+	H₂O →	R-NHCOOH> Carbamic acid	R-NH ₂ Amine	+	CO ₂	
R-NH ₂	+	R-NCO →	R-NHCONH-R _{Urea}				(1)

PU rigid foam with a closed-pore structure has applications in construction, refrigeration, and piping/tubing. PU foam density is in the range from 8 to 320 kgm⁻³, or in some cases higher [21]. PU rigid foam displays better thermal insulation than commercially available panels made of polystyrene (PS) or wool [5]. The self-adhesiveness of PU is also beneficial for producing a sandwichconfiguration composite, as it attaches to the board without other adhesives, on pouring a mix of raw materials into the cavity between the surface panels. Thin and flexible surface layers, such as paper or foil, enable continuous production by feeding these layers from rolls. However, due to their limited dimensions, it is impractical to apply thick wood-based surfaces such as particleboard or OSB (Oriented Strand Board). Therefore, the surface and core layers are prepared separately in the batch process.

The sandwich production process, patented by Lüdtke et al. [12], allows the continuous production of wood-based

surfaces and foam core layers in the same unit. Thermoplastic granulated foam has been used in this process by Lüdtke et al. [12] and Shalbafan [18]; this type of plastic requires a cooling phase for setting after forming the core layer. On the other hand, PU as a thermo set does not require this stage, which reduces operation time and cost. The raw components still need adjustments to perform satisfactorily as core materials: expandable under heat and activation temperature, allowing easy and uniform distribution. For this reason, the addition of solid filler into the liquid PU components is proposed to simplify PU formulation for application as core layer material.

Natural and synthetic fillers improve the structural and mechanical properties of PU foam, as reviewed by Gama et al. [6]. Applying lignocellulosic filler in polymers can be advantageous due to low density, economical production, renewability and biodegradability, CO₂ neutral character allowing combustion, and availability [1]. Wood fiber and flour were applied for

effects on foam properties in prior studies. literature [7, 24] Some confirmed improved properties with low wood contents, showing the possibility of applying wood as filler in PU foam. As regards participating in the reactions, Mosiewicki et al. [15] found that wood fiber increased the rate of gas loss during foaming and, accordingly, of matrix stiffness. Less expansion of PU could result from viscosity increase from the added wood flour in the formulation, degrading the compressive properties of PU foam as found by Krishnamurthi et al. [10] and Mosiewicki et al. [14]. It probably led to a more distorted structure of cells with a broader cell size distribution.

Besides using wood filler, the influence of heat treatment on PU foam properties is worth considering, for a core layer in lightweight wood-based panels with a density below 300 kgm⁻³. The PU foam core with particleboards showed good potential for use in the furniture industry [19]. Panel pressing at elevated temperatures aims at an incompressible and homogeneous core structure after expansion. A constant temperature is required to ensure a continuous reaction and foaming [9]. Regardless, elevated temperatures increase the reaction kinetics rates in PU foam [3]. Mohan et al. [13] concluded that the processing temperature significantly affected foam density and chemistry. The compressive strength parallel to the rise direction decreased at the normal condition of 23°C. Still, it grew at a temperature of -196°C, as proven by an increase in the molecular weight per branching unit of PU [20]. Even though several studies have dealt with the impacts of wood addition and the processing temperature on PU properties, both factors have rarely been

examined together. This work aimed to investigate the properties of PU rigid foam prepared with wood flour addition and thermal curing, to evaluate this combination for potential application in the continuous production of wood-based sandwich panels.

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2. Materials and Methods 2.1. Materials

The PU main components for rigid foam were provided by Covestro Company. Selected polyol (Desmophen 1240N) is a sucrose-based polyether polyol of high functionality (hydroxyl number = 445 mg KOH/g). For isocyanate, pMDI (Desmodur 44P16), di-/poly-isocyanate-component was used.

Surfactant is the only chemical additive used in rigid foam preparation. The silicone surfactant for PU/PIR insulating panels (Dow Corning 2938) was obtained from Dow Chemical Company.

Wood flour under the trade name Arbocel C100 was supplied by the J. Rettenmeier & Söhne GmbH and Co. KG Company. The material comprises raw cellulose with a grain size in the range 70-150 μ m. Screen analysis under DIN 734 by the supplier confirms the amounts of screen residue with an interior mesh aperture of 250 μ m being max. 0.5%, at 100 μ m being max. 45%, and at 32 μ m being max. 95%.

2.2. Polyurethane Production

Desmophen 1240N was dehydrated by heat before use. The wood flour was oven-dried at 103°C for 24 hr. Firstly, components with OH groups (polyol and water) were mixed, followed by surfactant and wood flour. After that, Desmodur 44P16 was added to the polyol mixture. The components were mixed by an electric hand mixer in aluminum cups, then placed into the oven for 15 min. Reacted foam samples were removed from the oven, then left at room temperature for at least 24 hr before the conditioning at 20°C with 50% relative humidity.

The wood flour contents of 0-12% in PU and the curing temperatures between 100

to 160°C were varied to determine their influences on foam properties, as presented in the following table with a total of 60 samples. The isocyanate (NCO) index measures the excess isocyanate used relative to the theoretical equivalent amount required for a 1:1 reaction with all active hydrogen. To calculate the NCO index in this research, the hydroxyl groups in the wood flour were not included.

Table 1

Sample	Wood flour content	Temperature	NCO	Water content	Surfactant
type	[% w/w _{total}]	[°C]	index	[pphp*]	[pphp*]
1	0	100	1.1	3	3
2	2	100	1.1	3	3
3	4	100	1.1	3	3
4	6	100	1.1	3	3
5	8	100	1.1	3	3
6	10	100	1.1	3	3
7	12	100	1.1	3	3
8	10	25	1.1	3	3
9	10	100	1.1	3	3
10	10	120	1.1	3	3
11	10	140	1.1	3	3
12	10	160	1.1	3	3

Controlled and varied parameters in the experimental design

*pphp: parts per hundred parts polyol

2.3. Preparation of Samples

Figure 1 shows the sample preparation of PUR foam. First, a cylinder-shaped sample of the PU foam was prepared by using the pillar drill in an aluminum cup, after the conditioning of the PUR samples. The test piece was then sectioned into three parts: the first largest sample with a thickness of 25 mm was used for the compression test. The second sample was further cut into at least six cubes for determination of open-cell content. The last sample was sliced into thin layers for microscopy analysis.

2.4. Characterization of Samples 2.4.1. Scanning Electron Microscopy (SEM)

The SEM-LEO1505 (Zeiss) scanning electron microscope was used to obtain micrographs of foam structure and to assess cell density features. In Lensdetector was applied with an acceleration voltage of 5.0 kV. Thin slices of foam specimens were cut into small rectangular samples and then glued on stubs to position the piece during the analysis. The specimen surface was coated with platinum before the imaging.



Fig. 1. Preparation steps of PU specimens for characterization of properties

2.4.2. Energy-Dispersive X-ray (EDX) Spectroscopic Analysis of Samples

SEM provided an elemental analysis by the EDX (Energy-dispersive spectroscopy) technique using an octane plus silicium drift detector (Octane plus SDD). The following diagram describes the chemical structure and ratios of the C, O, and N atoms in the raw materials. Their proportions are given as results from the EDX analysis.

2.4.3. Cell Size Measurement of Samples

The Keyence VHX5000 optical microscope was used to observe the surface morphology and to determine cell diameter (Figure 2). First, the foam sample was cut as thin as possible, then placed on a microscope specimen platform and examined optically using the following parameters: 250X magnification, light entry at 100% with ring illumination, and the observed area of 4-6 mm². 2Dpanorama images were created from 3D capture in all positions, and then processed by the provided software. Cell diameter was measured manually using the program from Keyence. The largest 50 cells were selected in each micrograph, where each cell's most extended and

shortest sides were measured for the calculation of average cell diameter.

2.4.4. Density of Samples

The samples of PU foam's density were selected from the specimens of compressive strength before testing. The density of the PU foam was decided by averaging the mass/volume measurement results of five specimens per sample following the procedure described in the ASTM D1622-98 standard [24].

2.4.5. Compressive Properties of Samples

compressive properties The were determined according to ISO 844. After the specimens were cut, they were conditioned at 20°C with 50% relative humidity until the test. The measurement was performed by using the universal Zwick Roell RetroLine testing machine including its software for data acquisition. The testing speed of the machine was set to 10% per min with a maximal loading of 0.5 or 5 kN, depending on the sample properties. The samples were placed in the compression testing machine and tested so that compression was parallel to the foam rise direction.



Fig. 2. Polyol and isocyanate structure modified from literature [21], cellulose structure [1] with calculation of C, O, N atom ratios in reference materials

2.4.6. Fourier Transform Infrared (FTIR) Spectroscopy of Samples

Neat PU and 10% wood-loaded PUR foam samples were analyzed using the FTIR-ATR (Bruker Vector 33, Mettler-Toledo GmbH) for assessing chemical structures. The FTIR analysis was performed with 2 cm⁻¹ resolution over the wavenumber range 600-3,800 cm⁻¹. The Opus 6.5 software was then used to evaluate the results.

2.5. Statistical Analysis

A one-way analysis of variance (ANOVA) was performed. The statistical differences between variations were evaluated by

multiple comparisons based on the least significant difference (LSD) test (Fischer's LSD test statistic). Statistical significance called for P < 0.05. All the data analysis was executed by using Microsoft Excel.

3. Results and Discussions 3.1. Chemical Properties of Samples 3.1.1. FTIR Analysis

Figure 3 shows the FTIR spectra of pure PUR and foam with 12% wood flour content. It can be seen that no new absorption peak arose from loading wood flour into the PU foam. This matches the FTIR results for olive kernel and nutshell reinforced PU [4]. The FTIR spectrographs of both samples evidenced urethane linkages (-NHCOO-) based on the reaction between polyol and isocyanate. In Figure 3, it is also clear that the sharp absorption peaks at 1,708.8 and 1,223.7 cm⁻¹ are seen. They correspond to

the stretching vibrations of -C=O and the asymmetric stretching vibrations of -C-O in urethane, respectively. Furthermore, the –NH stretching vibrations of urethane are observed at the wave number 3,320.1 cm⁻¹.



Fig. 3. FTIR spectra of heat-cured PU samples without and with 12% wood flour content. Absorbance peaks in bold numbers represent isocyanate, and those in a thinner font are for the polyol segments

The peaks in the wave number range 2,874-2,966 cm⁻¹ belong to the stretching vibrations of C-H in methyl or methylene. Also, the ether bond -C-O-C from polyol is confirmed by a broad and strong peak found at 1,070 cm⁻¹.

For the isocyanate segments, the absorption at 1,594 cm⁻¹ is caused by the stretching vibrations of C=C in the benzene ring. Several weak peaks are seen in 763-915 cm⁻¹ belonging to the bending vibrations of C-H in the multi-substituted benzene ring. Besides, the evident band at 2,276 cm⁻¹ relates to the unreacted isocyanate monomers [4, 8, 23].

3.1.2. Elemental Analysis

As illustrated in Figure 4, SEM images were selected to determine the elemental percentages. Two reference areas in Figures 4a and 4i represent PU surface and filler, respectively. Figure 4b-e displays SEM images with different filler distributions, divided into spotted PU surface (Figure 4b-e) and embedded filler in PU (Figure 4f-h).

The atomic proportions of the investigated areas are shown in Figure 5 compared to the calculated values from the reference materials. Of the three chemical elements, N is the only one that is unavailable in cellulose filler but is in isocyanate and in the pure PU matrix. It corresponds to the result of investigated areas in Figures 4a and 4i, which were related to PU and cellulose, respectively. The N atom percentage decreased slightly from 16.71% in PU > avg. 14.56% in spotted PU surfaces (b-e) > avg. 10.68% in areas with PU-embedded filler (Figure 4fh), while an increase in the percentage of O species was observed in the same order.



Fig. 4. SEM images of PU surface: (a) polymer surface, (b) to (e) spotted surface, (f) to (h) embedded filler with PU, (i) filler particle



Fig. 5. Percent contents of C, O, and N elements in the observed areas in Figure 4a-i shown together with the calculated values for reference materials

This finding confirmed the different distributions of cellulose flour in the polymer cells, in which a poor distribution tended to gather in the strut and appeared as having thick cell walls. On the one hand, a larger cell wall thickness can sometimes enhance the mechanical properties of the cellular material. On the other hand, it can be disadvantageous when associated with an irregular distribution. An embedded strut with high filler content constructs uneven cell walls and often leads to cell inhomogeneity, which can serve as a failure initiating defect in the structure under loading.

3.2. Physical Properties of Samples

According to Figure 6, the average cell size of PU mixed with 0-10% wood flour was within 244-397 μ m, which was significantly more than the 180 μ m cell diameter of conventional rigid foam according to Engels et al. [5]. Since wood flour with the grain size of 70-150 μ m was used in the study, these filler particles were large enough to interfere with cell development.

PU foam's cell size differed depending on the wood content. On adding a low amount of wood flour, as shown in the results with 2 and 4% wood content, both cell size and the width of cell size distribution decreased. Zhu et al. [25] also found that PU foams reinforced with fiber and nanoclays had smaller cell sizes than the neat PU foam. The fillers can serve as a nucleating agent for cell growth, resulting in an increased number of nucleated cells and decreasing the cell size in the reinforced foams. A decrease in the average cell size of PU foam with increasing wood filler content was also found in prior literature [10, 17]. No change in average cell size was observable with 6-10% wood flour contents. However, wood addition could expand the cell size distribution, particularly with 6% wood content. A wider cell size distribution is reasonable, as an increased mixture viscosity causes less expansion and accordingly causes a more distorted cell structure [14].



Fig. 6. Cell size distributions and averages (x) in PU foams with 0-10% wood content

The cell sizes in the samples produced at different temperatures are presented in Figure 7. In most cases, the average cell size in the PU foam formulations with 10% filler was consistently in 214-243 µm. An increase in reaction temperature from normal ambient temperature to 140°C did not raise the cell diameter significantly. Apart from water, heat affects the polymer's viscosity and the gas concentration dissolved into the matrix. Both of these control the foam growth process [11]. Despite the higher foaming rate at an elevated temperature, no change in the cell diameter was apparent.

A comparison of micrographs shows that PU foams cured at 120 and 140°C produced more anisotropic cells than the sample cured at lower temperatures that had consistent spherical cell shapes. This could indicate an accelerated reaction. As the PU reaction is exothermic, the generated heat transforms water into steam to help expand the foam. The reaction above the boiling point could increase the foaming rate due to the heat transfer from the evaporation, leading to an elongated foam structure and higher anisotropy.



Fig. 7. Cell size distributions and averages (x) in PU foams with 10% wood content cured at various temperatures

In Figure 8, the average density gradually increased with addition level of wood filler. The density remained practically unchanged with only slight differences for 0 to 2 and 4% cases. The density increase in wood-mixed PUR with

higher wood contents was probably due to the increased viscosity of polyol mixture with wood flour [24] and cell nucleation that increased the cell number density of foam [16].



Fig. 8. Density of PU foam samples

Not only did wood flour content impact the foam density, but also the reaction temperature. Foam samples prepared at room temperature without heat input had a significantly higher density than those cured by heat, estimated to be two-fold compared to results from 100° C, as shown in Figure 9. No significant differences in density were found among the heat-cured samples with various temperatures, as their bulk densities were on average from 48.03 to 69.17 kgm⁻³. Foam density decreased by the heat curing at 100-160°C, but no significant difference among the heat-cured samples was observed. Mohan et al. [13] also found that the processing temperature significantly affects PU chemistry and density. Based on their research, the average density decreased by 19% up to a curing temperature of 85°C, which was attributed to higher foam expansion resulting from decreased viscosity at higher processing temperature.



Fig. 9. Density of PU foam with 10 % wood content when cured at various temperatures

3.3. Mechanical Properties of Samples

As seen in Table 2, at up to 6% wood flour additions, the properties were at least similar or improved from the pure PU. Above this wood filler loading level, all compressive properties were reduced. However, there was no significant difference in compressive strength using a wood loading within 0-12% according to a statistical comparison between the groups with 0, 6, and 12%. The results confirmed the same trend as in a prior study [10], that the compressive strength decreased with increased wood flour content. That study explained the significant effect of filler content on compressive strength by: 1) moisture contents of the fillers; 2) generated initial CO₂ bubbles on the filler surface via the chemical reactions between isocyanate and water. The latter phenomenon can create many "tearing of continuity" locations at the particle-binder interface. These imperfections and defects in the physical microstructure caused a drastic drop in compressive strength.

Wood content	Compressive Strength [MPa]		Young's modulus [MPa]		Density [kg/m³]		Specific Strength [kN∙m/kg]
[70 W/W _{total}]	x	SD	x	SD	x	SD	x
0	0.22	0.05	2.44	0.65	48.05	4.34	4.62
2	0.23	0.04	2.53	0.5	45.98	3.03	5.07
4	0.26	0.03	2.95	0.53	48.47	2.27	5.29
6	0.25	0.04	2.58	0.47	53.99	4.83	4.63
8	0.17	0.01	1.77	0.12	50.47	2.47	3.46
10	0.20	0.02	2.24	0.47	54.73	0.98	3.57
12	0.17	0.04	1.91	0.41	56.90	5.50	2.96

Compressive strength of PU foams with different wood flour loadings Table 2

The overall trend showed a slight decrease in the strength-to-density ratio, namely specific strength, with rising filler content, according to Table 2. Yuan and Shi [24] found a similar result, namely that the compressive modulus of wood-PU foam did not decrease until higher wood flour contents of 20%, regardless of the increased density of samples [24]. The compressive properties for PU light foams were found to degrade with increasing natural filler content from 0 to 8% by Cabulis et al. [2]; in contrast, the current results show a noticeable reduction in properties starting from 8% filler content.

Table 3 presents the PU foam samples' compressive properties with 10% wood

content when prepared at different temperatures. The PU samples cured at elevated temperatures had properties degraded from those cured at the standard temperature. The compressive strength of the samples cured at 100°C became significantly reduced to less than one-third than that of the samples without heat curing, from 1.08 to 0.32 MPa. Considering the heat-cured PU samples, the results did not suggest a dramatic further difference. The compressive strength dropped from 0.32 to 0.14 MPa on elevating the cure temperature from 100 to 160°C.

Table 3

Curing Temperature	Compressive Strength [MPa]		Young's modulus [MPa]		Density [kg/m³]		Specific Strength [kN·m/kg]
[0]	x	SD	x	SD	x	SD	x
Normal	1.08	0.15	14.50	2.76	145.62	11.67	7.38
100	0.32	0.04	4.17	0.84	70.13	4.37	4.41
120	0.23	0.03	2.81	0.65	66.02	3.96	3.53
140	0.21	0.03	2.41	0.53	58.41	4.41	3.49
160	0.14	0.02	2.01	0.41	48.03	3.84	2.95

Compressive properties of PU foams with 10% wood content cured at different temperatures

Young's modulus showed a similar change rate affected by raising the curing temperatures. As the curing temperature was increased, all the compressive properties of the samples declined. Similar changes in the compressive properties were found previously [13, 20]. Even though heat influences foam development, the cell size of PU produced at standard and elevated temperatures was comparable. As discussed earlier in the section on cell size, the heat did not significantly induce cell size changes; however, it increased the foaming rate. Possibly the various curing temperatures caused changes in cell morphology, e.g. in the cell wall thickness or the cell number density, affecting foam structure and cell alignment, and thereby the response to mechanical loading.

Previous publications [24, 25] have confirmed the relationship between

compressive properties and cell size and density of PU foam. The mechanical properties and the material density are related in a simple power law in the work by Yuan and Shi [24], as follows: where A is a constant related to the temperature and physical properties of the resin, and B is related to the deformation mechanics of the cellular materials.

The compressive modulus of the samples with four different wood contents is related to their densities in Figure 10.



Fig. 10. Relationship between compressive modulus and density for PU foam with different wood contents



Fig. 11. Relationship between compressive modulus and density for foam cured at different temperatures (0-160°C). PU samples cured at room temperature are shown with filled square symbols

the work deformation

 $\log(\text{strenghtproperty}) = \log A + B \cdot \log(\text{density})$

Figure 11 shows the dependence of compressive modulus on density among PU samples using different curing temperatures. Despite the alternative curing temperatures, PU with 10% wood flour had the same dependence of compressive properties on density as in the last figure with the same curing temperature. The results show a higher density exponent than the last one, now at 1.89, which resulted from higher compressive modulus and density of PU cured at standard temperature [24]. Since the specific strength of the PU sample group cured at room temperature was 7.38 kN·mkg⁻¹, it amounted to 1.7-2.0, proportionally higher than the values for the PU groups cured by high temperature.

4. Conclusion

FT-IR spectra were not significantly different between neat and 12% woodcontaining PU foam. A higher wood content leads to a wider distribution of cell sizes in the PU foam. With a small proportion of wood filler (2 or 4%), cell size decreased on average due to the filler functioning as a nucleating agent, increasing the number density of the cellular pores. The measurement of the compressive properties confirmed a meaningful effect of higher wood contents past 8% filler content. The compressive strength of PU foam with 8-12% wood flour was approximately 0.175 MPa, which is approximately 2 % lower than that with 0-6 % filler loading.

On using ambient curing temperature, more homogeneous cell shapes with a smaller average size could be found. A remarkably higher foam density was found for PU cured at this standard temperature than in samples cured at 100°C, with a 50% reduction in density after the elevated curing temperature was applied. Regarding the similarity in cell size between both groups, it can be assumed that PU without heat curing has a thicker cell wall or strut than with the other treatments. Its higher consistency leads to the highest compressive properties superior to the heat-cured samples. While 1 MPa compressive strength was obtained for PU cured at temperature, room the heat-cured samples exhibited compressive strengths averaging only 0.2 MPa. The decline also appeared in Young's modulus and in specific strength. The compressive properties fall when increasing the curing temperature because of higher anisotropy and inhomogeneity in cell shapes, induced by faster reaction kinetics at higher temperatures. However, the possibility of improving the characteristics of the PU core used for sandwich structures by raising the temperature and adding wood flour content needs further study for improving the properties of such a green composite material.

Author Contributions

The manuscript was written with contributions from all authors. All authors have given their approval to submitting the final version of the manuscript.

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Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

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