Tackiness of a novel biodegradable binder for erosion control mulch

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ABSTRACT

Kraft lignin, acidic (LA) and alkaline (LB) types, byproducts of pulp manufacturing were mixed with crude glycerol, a by-product of biodiesel production, and humic acid to produce a mulch tackifier for soil erosion control. Lignin was dried at 70°C for 24 h before use. The study was a full factorial with lignin levels of 20, 50, and 80% and humic acid levels of 1, 2, and 5% (w/w). The remaining portion consisted of crude glycerol. The samples were homogenized using water to facilitate the homogenization process. After homogenization, the samples were left to settle for 36 h and then separated into solid and liquid portions. Both portions were dried at 70°C for 24 h and tested for tack after wetting with water. Dried lignin, as a standalone ingredient, was also tested for tack. The values for the strength of tack were compared to those obtained from a commercial tackifier. Acidic lignin could be used with lignin content ≥50%. Acidic lignin was comparable to the commercial tackifier, whereas alkaline lignin was found to have significantly more tack but was found to be unsuitable for soil erosion use.

RÉSUMÉ

Des types acide (LA) et alcalin (LB) de lignine Kraft, sous-produits de l'industrie des pâtes et papiers, ont été mélangés à du glycérol brut, un sous-produit du biodiésel, et à de l'acide humique pour produire un paillis collant pour le contrôle de l'érosion du sol. La lignine a été séchée à 70 °C pendant 24 h avant son utilisation. L'étude factorielle complète a été réalisée avec des niveaux de lignine de 20, 50 et 80 % et des niveaux d'acide humique de 1, 2, et 5 % (p/p) ; le glycérol brut constituant la portion restante. Les échantillons ont été homogénéisés avec de l'eau pour faciliter ce processus. Après l'homogénéisation, les échantillons ont reposé pendant 36 h et ont ensuite été séparés en portions solide et liquide. Ces deux portions ont été séchées à 70 °C pendant 24 h et testées pour leur propriété adhérente après avoir été mouillées avec de l'eau. La lignine séchée, comme ingrédient, a aussi été testée pour sa propriété adhérente. Les valeurs de cette propriété ont été comparées avec celle obtenue d'un adhérent commercial. La lignine acide pourrait être utilisée à un niveau excédant 50 %. La lignine acide était comparable à l'adhérent commercial tandis que la lignine alcaline s'est avérée être plus adhérente, mais moins adaptée au contrôle d'érosion des sols.

KEYWORDS

Lignin, humic acid, glycerol, tack, tackifier, erosion control

MOTS CLÉS

Lignine, acide humique, glycérol, adhérence, agent adhérent, contrôle de l'érosion

INTRODUCTION

Soil erosion is a naturally occurring process involving three distinct phases: soil detachment, movement, and deposition (Ritter 2012). Soil erosion control is an attempt to put a process in place to achieve several objectives: to slow the soil erosion process to a tolerable level, to stop it, and to rebuild soil vegetation and organic matter. Soil erosion control seeks to support the formation of stable aggregates within which soil particles are sufficiently adhered to each other to provide resistance to being washed away. An additional objective of soil erosion control is to form adequate macropore space to allow water to move vertically through the soil rather than along the gradient of the slope. Additionally, resistance to erosion increases with higher soil shear strength, which can be obtained by interlocking aggregates, increased friction, and stronger attractive forces between soil aggregates.

Lignin has been researched as a soil erosion retarder (Shulga et al. 2001; Shulga et al. 2007; Shulga et al. 2008). Lignin is insoluble in water, in most organic solvents, and generally, in acids, but is soluble in bases (Tan 1998). Due to its recalcitrant nature, lignin tends to accumulate in soils and is considered an essential source for soil humus formation and especially humic matter. Lignin reacts with ammonia making it a carrier for nitrogen fertilizers (Tan 1998). Zhang et al. (2004) found that the addition of lignin to soil had positive effects on soil pH, organic matter (OM), cation exchange capacity (CEC). Moreover, lignin increased the carbon, nitrogen, phosphorous, and potassium levels. Adding lignin to soil resulted in a significant increase in biomass and shoot height in winter wheat and a substantial decrease in the accumulation of heavy metals in the wheat shoots.

Glycerol is a byproduct of biodiesel production. For every 10 kg of biodiesel produced, about one kilogram of glycerol (crude) is generated (Jitrwung and Yargeau 2015). Glycerol can be applied with water as a foliar spray or by drench application, with positive effects on plant growth (Tisserat and Stuff 2011). Foliar application produced better results than root drenching. In other studies, it has been noted that applying glycerin (a commercial term for glycerol) in concentrations from 0.1 to 10% to plant roots before shipping and transplantation decreased mortalities and incidence of stem canker. When plant roots were treated with glycerin, the plants started growing sooner than those treated with water. When bulbs are soaked in glycerin solutions as low as 0.1%, just before planting, the yield was much higher than bulbs soaked in water.

Glycerol is a highly hygroscopic humectant (National Center for Biotechnology Information 2020), attracting and retaining moisture under adverse conditions. As a soil amendment, it may prove beneficial beyond that of adding carbon to the soil. At lower rates (0.1 and 1 kg/ha), glycerol can be an effective soil amendment resulting in increased crop yield. However, high application rates (10 t/ha) immobilize nitrogen and phosphorous (Qian et al. 2008). Glycerol, when applied together with nitrogen fertilizer, significantly increased organic content in the soil. The addition of nitrogen fertilizer to glycerol application ensures that plants and microorganisms have adequate access to available nitrogen (Qian et al. 2011).

Humic acids are water-insoluble stable products of decomposed biomass. Humic acids improve cell membrane permeability and facilitate a higher uptake of ions in root tissue for enhanced plant growth. Moreover, humic acids stimulate the growth of autotrophic nitrifying bacteria (Valdrighi et al. 1996). For the current study, humic acid is proposed as an emulsifier of lignin in glycerol. The additional properties of humic acid as a surfactant and carrier of plant growth hormones may provide benefits besides soil erosion control.

To the knowledge of the authors, there has been, to date, no attempts to produce a soil erosion tackifier combining the three ingredients; lignin, glycerol, and humic acid. This study is based on the hypothesis that a combination of these ingredients will form the basis for a novel tackifing agent that may prove, in future studies, to be a useful agent for soil erosion control purposes.

Cohesion is the property of internal bonding between particle surfaces of homogeneous composition. Adhesion is the property of bonding between heterogeneous surfaces. Tack is the property by which pressure-sensitive adhesives (PSA) form bonds without the use of any solvent, dispersant or heat source (Creton and Fabre 2002). Tack is a complex mechanism. The traditional method for studying tack and tackiness has been to correlate tack with the rheological behaviour of the adhesive (Bikerman 1947). Bikerman (1947) describes tackiness as "The resistance which must be overcome to separate two solids joined by a liquid adhesive" Whereas tack is technically defined as the property of the mechanism of the formation of the bond, the tackiness is the measurable resistance of separating that bond. The two terms, tack and tackiness, are often interchanged though it is understood that there is no tackiness without tack. When tackiness is measured in terms of the energy to break the bond, the term 'tack energy' is often used. Creton and Fabre (2002) contend that although useful information can be found through examining tack as a rheological behaviour, this approach is overly simplistic. Measured tack energies are much larger than the thermodynamic work of adhesion. Three conventional methods used to test tack are the probe method, the rolling ball or cylinder, and the loop tack test. The ASTM International method for testing the peel or stripping strength of adhesive bonds is a modified version of the loop tack test (ASTM D903-98 2010). The loop tack and rolling ball or cylinder are easier to implement than the probe method. However, the probe method gives more insight into the tackiness of the adhesive (Creton and Fabre 2002). Increased viscosity leads to a more substantial cohesive strength, manifesting itself in more robust, more stable fibrils when the bonded surfaces are forcibly separated (Creton and Fabre 2002). For tack to be measured, the adhesive must bond to two surfaces through adhesion, and

when those surfaces are pulled apart, the cohesive bonds in the adhesive must break while a portion of the adhesive remains on each surface.

The primary objective of this study is to test the hypothesis that crude glycerol, Kraft lignin, and humic acid can be combined to form a tackifier for erosion control purposes. The sub-objectives of this study were 1) to determine whether acidic (LA) or alkaline (LB) lignin would provide better values for tack, and 2) to elucidate a range of ratios of glycerol, lignin, and humic acid for said tackifier that meet specific criteria, showing potential for commercial use. The criteria that would define said tackifier for commercial potential are forming a homogeneous dry powder that demonstrates tack when wetted with water.

MATERIALS AND METHODS

Materials

Glycerol Crude glycerol, a byproduct of biodiesel production, was provided by ECBVerdyol, Riverton, MB. The catalyst used for the initial biodiesel production was potassium hydroxide (KOH). The pH of the glycerol was measured without water dilution using an electronic pH meter. No attempt was made in this study to analyze the ratios or any of the components generally associated with crude glycerol. The impurities present in crude glycerol, obtained from biodiesel production, were assumed to have an insignificant effect on the crude glycerol's ability to act as a plasticizer or a wetting agent. The glycerol was stored in a plastic five-gallon pail at room temperature and was stirred before using, and samples were representative.

Lignin Lignin samples were obtained by ECBVerdyol from Hinton Pulp, a division of West Fraser Mill Ltd., Hinton, AB. Two Kraft lignin forms were used: lignin A wet cake (LA) and lignin B wet cake (LB). A certificate of analysis was provided with the samples for which the quality analysis portion is summarised in Table 1.

Both LA and LB were dried in a convection oven at 70°C for 24 h using thin-layer drying and manual stirring about every 30 min for the first four hours. Initially, the samples were dried at 100°C, but it was found that at this temperature, the sample (LB) tended to cure or set much like a wet cement powder, and an odour much resembling charring wood was given off. The temperature was lowered to 70°C, which produced a dry sample without curing or releasing an odour. The dried samples were stored in a desiccator at room temperature until needed. Dry LA and LB were tested for pH using an electronic pH meter by mixing one lignin with ten parts water (w/w).

Table 1. Quality analysis for lignin A wet cake and lignin B wet cake.

|	Unit of measure	Test Results	
		Lignin A	Lignin B
% Solids	% (w/w)	47.5	57.1
% Ash	% (w/w) dry basis	0.96	26.52
pH	pH	4.4	10.3

Humic acid Humic acid (HA) with the product name, K Hume Activated Humate, was supplied by Mesa Verde Humates through their Canadian distributor Green Chem Consultants Inc. The humic acid was crushed with a pestle and mortar to a fine powder before using it. K Hume is reported as 70% HA activated potassium humate.

Water Water used for the samples was Winnipeg tap water with no additional filters or treatment.

Sample Preparation

Samples were prepared by combining portions, by mass, of lignin (either LA or LB), HA, and glycerol to nine ratios as per Table 2. The proportions were selected to give insight into workable ranges as per the second sub-objective of this study. For samples containing LA, the total sample size was 120 g. The dried lignin was weighed and placed into a tared straight-sided glass jar 16 oz (473 mL) from Uline, (Milton, ON). Humic acid was weighed and crushed into a fine powder using a pestle and mortar and then added to the lignin jar. Glycerol was weighed and added to the jar with the lignin and humic acid. Homogenization and wetting with crude glycerol were not achievable without innovating a mixing device. Two parts water for one part mixture was added to the jar to facilitate homogenization. The mixture was homogenized using an Ultra-Turrax homogenizer Model number SD45, Serial number 11559, from Janke & Kunkel KG IKA-Werk (Germany). The authors designed a paddle blade and manufactured in-house to fit the homogenizer (Figure 1).

During homogenization, excessive foaming hindered adherence to a pattern of intervals of homogenization and resting. Therefore, the samples were transferred to larger containers to allow for more continuous mixing. However, the problem of excessive foaming remained, and homogenization was completed by mixing until the foam reached near the top, then waiting for the foam to settle and subsequently repeating these two steps until a homogeneous mixture was achieved. The resulting mixtures were suspensions. The samples were allowed to
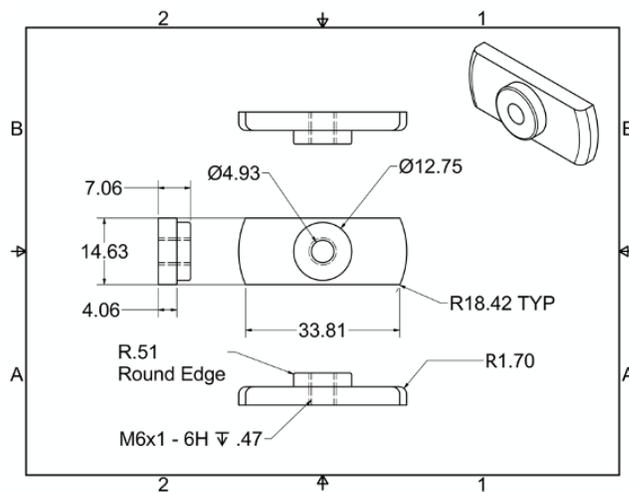
Fig. 1. The paddle blade for the homogenizer (mm).

settle for 36 h during which a solids fraction settled to the bottom, and a liquid fraction remained on top. The liquid portion was poured into an empty jar. Both parts were dried at 70°C (24 h for the solids portion or until dry for the liquid portion).

Lignin B was mixed in the same manner as LA with the following adjustments. The total sample size was reduced from 120 g to 100 g to avoid the necessity of transferring the mixture to a larger container for homogenizing. However, foaming was more excessive with LB than with LA and the need to transfer to a larger container remained. Additionally, LB did not wet as readily as LA, so that LB homogenization required an additional step of manually shearing the different portions between two metal spatulas to obtain a homogeneous mixture. Upon reaching a homogeneous mix, the samples were settled, separated, and dried as per samples containing LA. Lignin B samples with 80% lignin formed a colloid. The colloid mixtures were dried at 70°C for 24 h.
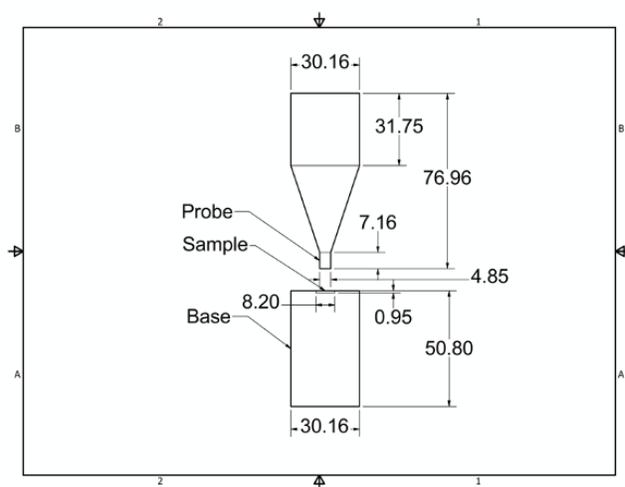
Dried samples that did not resemble a fine powder were crushed using a pestle and mortar, and all resulting powders were retained in glass jars at room temperature until a probe tack test was done.

Samples for the probe tack test were prepared by placing a small portion (about 1 g) of the dried samples in a Petri dish with water at a ratio of 1:1 (w/w) and mixed with a metal spatula and allowed to equilibrate for 3.5 h and after that tested for tack. Dry lignin as a standalone ingredient, including LB samples with 80% lignin content, were also tested for tack using ratios of 1:1 and 2:3, parts mixture to parts water, respectively.

As a reference point, Nilex T300 (a commercial tackifier) was mixed in three ratios with water: 1:10, 1:5, and 3:10 parts T300 to parts of water (w/w) and tested for tack (Table 2). Three ratios of mixing Nilex T300 with water were used because the product's tackiness is dependent on wetting, and the authors wanted to understand how adding water might influence the tackiness of the product.

Fig. 2. Probe and base with sample outline (mm). The probe comes down into the sample then pulls up to measure tack. An example is placed in the counterbore in the base diameter 8.20 mm by 0.95 mm deep.

Sample Testing

Sample testing was done using a probe tack test. A probe and base were designed and manufactured in-house to fit an LS5 Universal Test Machine from Ametek Inc. (Largo, FL). The probe and base were made of stainless steel (Figure 2).

Before testing, the probe was lowered into the counterbore in the base at a rate of 1 mm/s until a force of 0.25 N was attained, and this position was defined as the bottom. The probe was raised, to the test starting point, 1.5 mm above the bottom. A portion of the sample was placed into the counterbore in the base using a metal spatula. The counterbore was filled by lightly pressing the flat end of the spatula over the sample. The spatula was scraped along the face of the base to smooth out the sample making it flush with the top face of the base. The steps of pressing the

Table 2. Ratios of materials for testing.

Mix Name	Percentages by (w/w)			Part by (w/w)		
	Lignin	Humic acid	Glycerol	Lignin	Tackifier	Water
L(A or B) 20 HA 1	20%	1%	79%			
L(A or B) 50 HA 1	50%	1%	49%			
L(A or B) 80 HA 1	80%	1%	19%			
L(A or B) 20 HA 2	20%	2%	78%			
L(A or B) 50 HA 2	50%	2%	48%			
L(A or B) 80 HA 2	80%	2%	18%			
L(A or B) 20 HA 5	20%	5%	75%			
L(A or B) 50 HA 5	50%	5%	45%			
L(A or B) 80 HA 5	80%	5%	15%			
L(A or B) 1W1				1		1
L(A or B) 2W3				2		3
Nilex T300 1 to 10					1	10
Nilex T300 1 to 5					1	5
Nilex T300 3 to 10					3	10

sample and scraping provided a homogeneous sample. To gain insight into the behaviour of the mixtures, a probe tack test of five repetitions was performed at four stages of product development: 1) after the settling period of 36 h on the solids portion, before drying (denoted with suffix 'orig') 2) on the dried solids portion mixed with an equal part, by mass, of water (denoted with suffix 'F'), as well as, 3) on the liquid portion, after drying off the water, also mixed with an equal part, by mass, of water (denoted with suffix 'W'), and 4) on the liquid portion after drying with no water added (denoted with suffix 'wet'). A fifth probe tack test was done on all LB samples, at the second stage, with 80% lignin content mixed with water at a ratio of 2:3 lignin to water, respectively.

The probe tack test consisted of two steps: 1) lowering the probe into the sample at a rate of 1 mm/s until a force of 20 N was reached, 2) pulling the probe up at a rate of 10 mm/s to a height of 6 mm above the starting point. The test direction was set to compression so that forces pushing the probe into the sample were recorded as positive. The strength of tack restricting the upward movement of the probe was recorded as negative. The test frame interface was programmed to record one thousand evenly spaced data points, including probe position, time, and force. Additionally, the minimum force encountered throughout the test was recorded as a single data point.

Data Analysis

Analysis of data was automated using Python 3.5.2 (Python Software Foundation 2016) with the StatsModels library 0.8.0 (Seabold and Perktold 2010) and Pandas 0.23.0 (McKinney 2010). The absolute value of the minimum force (N) encountered in the probe tack test was divided by the constant area of the probe ($A = 18.47$ mm^2) to quantify the tack's strength. As the area remained constant, the additional step of dividing the force by the area was eliminated, and the value of the minimum force was used for comparison purposes. Results were analyzed using an analysis of variance (ANOVA) with a pairwise Tukey Honest Significant Difference (HSD) test to identify significant differences between samples.

RESULTS AND DISCUSSION

Materials

Lignin and Glycerol pH Dried lignin samples tested for pH returned values of 5.3 and 10.5 for LA and LB, respectively. These values correspond closely to those provided by the manufacturer. Glycerol pH was found to be 8.7, which is reasonable for a liquid with KOH.

Procedure for Sample Preparation

Each of the two lignins, LA and LB, reacted uniquely to the sample preparation procedure. For LA, only samples with ≥50% lignin (w/w) and LB samples with ≥80% lignin (w/w) produced products that met the criteria of the second sub-objective of this study. Humic acid proportions profoundly influenced the three LB 80% samples' viscosity, with a positive correlation between the HA portion and viscosity (data not reported).

PROBE TACK TEST

Lignin A Of the four stages in the sample preparation, only the dried solids portion mixed with water provided a useful measure of tackiness (suffix 'F'). For the dried solids portion, the only sample that was significantly different from all the other samples was LA wetted with water 1:1 (w/w) (sample name LA1W1, Table 2).

Lignin B The probe tack test performed before the drying stage showed significantly increased tack corresponding to higher lignin content and a significant influence corresponding to humic acid contents. The values for 5% humic acid content with 80% LB were insignificantly lower than those with 1 and 2% HA. However, it should be noted that the viscosity and level of cohesion were visibly much higher in the colloid that formed from 80% LB and 5% HA and that on several probe tack tests, the cohesion was so high that the entire sample adhered to the probe. In these cases, the whole sample pulled out of the base without breaking any cohesive bonds. Generally, increased viscosity is associated with increased tack; however, when viscosity levels get too high, tack is lost as the material loses its ability to create fibrils. This appears to have happened with the LB 80 HA 5 samples (Creton and Fabre 2002). This observation leads to the postulation that when diluted with water, as would be the case in field application, this mixture could outperform those with less HA content.

The mean values for force (Table 3) were used to measure the strength of tack for both lignin types that produced viable end products. The three mixtures of the Nilex T300 commercial tackifier were analyzed using an ANOVA table. They were found to be significantly different from each other. The Tukey HSD-pairwise-comparison test revealed that, except for LA1W1, LA samples did not differ from each other or Nilex T300. However, the strength of tack values for LB samples, except for LB1W1, was significantly higher than all LA and Nilex T300 samples. Except for LB1W1, LB samples did not significantly differ from each other (Table 3).

After the completion of this study, it was found, in parallel studies concerning the use of LA and LB in other novel products, that LB tends to solidify, in transport, ruining the transport vessel and making its use impossible. Moreover, LB is high in sodium bicarbonate, making its use for agricultural purposes questionable (Lake 2018, personal communication). Thus, LA for soil erosion control provides the optimal choice with the strength of tack similar to Nilex T300.

It is recommended to conduct further testing on LA with ≥50% lignin content. Areas of recommended research include 1) cost analysis, 2) the effect on germination and rooting for plant seeds used with hydroseeding applications, 3) humectant properties, 4) optimal moisture content range for sustained tack, and 5) the interaction of the proposed product with various mulch mixes. Of interest for study would also include alternative humic acid products together with LA.

Table 3. Values for the strength of tack, n = 5, superscript a to e indicate significant groupings using a Tukey Honest Significant Difference pairwise comparison. Means with different letters are significantly different. Critical Value = 1.10. α=0.05.

Name	Suffix	Mean (Force N)	SE
LA1W1		0.17^a	0.08
LA 80 HA 2	F	0.69^{ab}	0.08
Nilex T300 1 to 10		0.74^{ab}	0.03
LA 50 HA 2	F	0.84^{ab}	0.05
LA 50 HA 1	F	0.87^{ab}	0.24
LA 80 HA 5	F	0.94^{abc}	0.12
LA2W3		0.98^{abc}	0.08
LA 80 HA 1	F	1.01^{abc}	0.09
LA 50 HA 5	F	1.16^{abc}	0.15
Nilex T300 1 to 5		1.58^{bc}	0.07
Nilex T300 3 to 10		1.60^{bc}	0.15
LB1W1		1.98^{cd}	0.38
LB 80 HA 5	F	3.03^{de}	0.26
LB 80 HA 5	F2W3	3.40^e	0.28
LB 80 HA 2	F	3.47^e	0.33
LB 80 HA 1	F	3.48^e	0.26
LB 80 HA 1	F2W3	3.50^e	0.36
LB 80 HA 2	F2W3	3.58^e	0.32
LB2W3		3.85^e	0.10

This study demonstrated that glycerol was an insignificant factor for tackiness, within the boundaries of mixtures tested. This observation suggests that LA added to mulch, without a drying step, may suffice for a tackifier suitable for soil erosion control. An alternative approach would be to add the lignin and the humic acid, without drying, to the mulch, and to add glycerol with the water during the hydroseeding premixing stage.

CONCLUSIONS

Lignin types A and B were mixed with HA and glycerol in ten combinations of ratios respectively: 20:1:79, 20:2:78, 20:5:75, 50:1:49, 50:2:48, 50:5:45, 80:1:19, 80:2:18, 80:5:15, and 100:0:0. It was found that both lignin types were able to produce a tackifier with strength qualities comparable to or greater than those for Nilex T300, a commercial tackifier. Samples from LA with ≥50% lignin content yielded strength of tack values ranging from 0.17 – 1.16 N comparable to Nilex T300 with the strength of tack values ranging from 0.74 to 1.60 N. Samples from LB with ≥80% lignin content produced strength of tack values (min = 3.03 N) significantly higher than those from Lignin A and Nilex T300 (max = 1.60 N) (critical value = 1.10 N).

Through personal communication with other researchers, it was also discovered that LB would not be a right candidate for a tackifying product, for soil erosion control, for reasons beyond the scope of this study.

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