

Comparison of treatments for cellulose pulp from agro-industrial wastes from the Amazon region

Comparación de tratamientos para pasta de celulosa de residuos agroindustriales de la región amazónica

Grober Panduro-Pisco (D^{1*}, Angie Stefani Amasifuen-Rengifo¹, Edinson Rubina-Arana (D¹, David León Moreno (D¹)



¹Facultad de Ciencias Forestales y Ambientales, Departamento de Conservación de Recursos Naturales, Universidad Nacional de Ucayali. Carretera Federico Basadre km 6, Pucallpa-Perú.

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Amazonía; biomasa; compuestos de agua destilada; Sal de Mohr **ABSTRACT:** Agroindustrial waste (AIW) is a potential source of cellulose, which can be obtained through different treatments. In this study, we evaluated four delignification treatments (10% sodium hydroxide, 50% ethanol, distilled water, and 25% Mohr's salt) to obtain cellulose pulp from four Amazonian AIWs (banana peel, cassava peel, sugarcane bagasse, and rice husk). Our results showed that sodium hydroxide treatment had the highest lignin removal and increased cellulose content, while Mohr's salt treatment had the lowest cellulose yield and lignin removal. Banana peel and rice husk had the highest cellulose yield, while cassava peel had the lowest. Distilled water treatment at medium temperature had similar lignin removal and cellulose yield to the sodium hydroxide and ethanol treatments. Our findings suggest that AIWs have great potential as a source of cellulose and that these economical, simple, and eco-friendly treatments can be used to obtain high-purity cellulose from AIWs.

RESUMEN: Los residuos agroindustriales (RAI) son una fuente potencial de celulosa, que puede obtenerse mediante diferentes tratamientos. En este estudio, evaluamos cuatro tratamientos de deslignificación (hidróxido de sodio al 10%, etanol al 50%, agua destilada y sal de Mohr al 25%) para obtener pulpa de celulosa a partir de cuatro RAI amazónicos (cáscara de banano, cáscara de yuca, bagazo de caña de azúcar y cascarilla de arroz). Los resultados muestran que el tratamiento con hidróxido de sodio tuvo la mayor remoción de lignina y aumentó el contenido de celulosa, mientras que el tratamiento con sal de Mohr tuvo el menor rendimiento de celulosa y remoción de lignina. La cáscara de plátano y la cáscara de arroz tuvieron el mayor rendimiento de celulosa, mientras que la cáscara de yuca tuvo el menor. El tratamiento con agua destilada a temperatura media tuvo una eliminación de lignina y un rendimiento de celulosa similares a los de los tratamientos con hidróxido de sodio y etanol. Nuestros resultados sugieren que los RAI tienen un gran potencial como fuente de celulosa y que estos tratamientos económicos, sencillos y ecológicos pueden utilizarse para obtener celulosa de gran pureza a partir de RAI.

1. Introduction

Globally, approximately 100 billion metric tons of agroindustrial waste (AIW) is generated each year [1]. Much of this waste comes from industrial activity; its management, use, and disposal represent a major challenge in developing countries [2, 3]. In the Amazon

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* Corresponding author: Grober Panduro-Pisco E-mail: grober_panduro@unu.edu.pe ISSN 0120-6230 e-ISSN 2422-2844

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region, the increase in agro-industrial activity since the 1960s [4] has resulted in a constant and growing generation of this type of waste. The Ucayali region in Peru is highly agro-industrial, with the main cultivated crops being oil palm, *palmito*, cacao, sugar cane, rice, *camu camu*, cavassa and banana. In most Amazonian cities of Peru, there is a limited application of feasible and efficient technologies to recycle these wastes and convert them into products that could improve environmental quality. This AIW has a high potential for use due to its diverse chemical composition. When AIW is not disposed of properly, it can cause serious health and environmental problems. Much of the biomass waste is left in the field to decompose naturally, taken to landfills or dumps, or subjected to thermal processes (*i.e.*, incineration, burning, or charcoal production). These processes, besides being inefficient, generate greenhouse emissions that deteriorate air quality [2]. Hence, the importance of developing effective and eco-friendly strategies to manage this type of waste.

Fruit and vegetable peel in AIW are very significant. As most of the peels are discarded as waste, they are not reused and represent a serious disposal problem [5]. Sugarcane bagasse and rice husks are among the most abundant AIW; with their annual global generation estimated to be 181 and 110 million tons, respectively [6]. AIW can be reused for obtaining energy (*i.e.*, bioethanol, biodiesel, biogas, etc.), composting, animal feed production, elaborating construction material, and implementing environmental remediation strategies [7]. However, in recent years, there has been extensive research on biomass residues to extract and add value to polymers such as cellulose, lignin, collagen, keratin, and chitosan [2].

Bananas are tropical fruit consumed worldwide, and there are many varieties. The banana peel is the main residue and represents between 30 and 40% of the wet weight of the fruit. This residue has been used mainly used in composting, as animal feed, and in the production of ethanol, enzymes, methane, proteins and pectins. The banana peel is mainly composed of cellulose, hemicellulose, pectin, and chlorophyll [5].

Similarly, cassava is one of the most important products in countries such as Indonesia, or Nigeria. Cassava is used as raw material for cassava starch production and culinary use. Its leaves can be used as a vegetable or as a natural medicine due to the large number of proteins and other bioactive compounds it contains. The woody part of the plant is used as cooking fuel. The processing of cassava starch generates large amounts of waste, including cassava peels [5].

In contrast, sugarcane is one of the most widely grown crops in tropical and subtropical countries worldwide. Sugarcane is used for juice extraction and sugar production. About 80% of the world's sugar demand is met by sugarcane cultivation. The main residue after processing is sugarcane bagasse, usually generated after cleaning and juice extraction [8, 9].

Finally, rice is an essential food grown in more than 75 countries in the world. Every year, about 80 million

tons of rice husks are generated as waste. Rice husk is the outer layer of rice; it is generated as a by-product of milling and accounts for about 20% of the fresh weight of total rice produced [10].

AIW is a natural source of polymers that are non-toxic, biodegradable, and biocompatible, unlike fossil-resources derived polymers. Cellulose, which is one of the most abundant polymers, is widely used in the paper industry [2]. It accounts for approximately 40-50% of plant and woody biomass by weight and exhibits high strength while being renewable and biodegradable. Cellulose is used in various industries, such as textiles, paper, materials, food, and pharmaceutical, and chemical industries. Obtaining cellulose maximizes recycling and minimizes waste [11].

There are numerous technologies to extract cellulose from biomass, classified as physical, chemical, physicochemical, and biological [11]. Sodium hydroxide is an alkaline chemical treatment and is widely used for lignin removal from biomasses [12, 13]. Ethanol is a preferred compound for organosolv chemical treatments, that employ organic compounds at high temperatures [14]. Other authors have used organic compounds at low temperatures to remove lignin. For example, Wi *et al.* [15] removed lignin from various materials using hydrogen peroxide–acetic acid at 80°C, and Reales *et al.* [16] evaluated lignin removal with an organosolv treatment at 60°C.

However, these treatments have the disadvantage of employing chemical compounds and producing hazardous liquid waste during the process. Therefore, Mohr's salt and distilled water were selected as the other two treatments based on the premise that they do not use hazardous chemicals and are eco-friendly. Mohr's salt has been successfully used for paper production from sugarcane leaves [17]. Hot water methods, as well as alkaline and organosolv treatments, involve the use of pressure and high temperatures [12]. Lower treatment temperatures and shorter treatment periods contribute to reducing production costs [18, 19]. Based on the principle of hot water treatments, distilled water at medium temperature was studied as a possible treatment for delignification.

This study addresses the research gap for more environmentally friendly and sustainable methods for obtaining cellulose from agroindustrial waste. While previous studies have explored various treatments for cellulose extraction, this research focuses specifically on delignification treatments and their potential for lignin removal and cellulose yield from a specific set of agroindustrial waste samples. Furthermore, this study evaluates the potential of simple and eco-friendly technologies that can be used in resource-limited settings to obtain high-purity cellulose from waste materials, contributing to the development of sustainable solutions for waste management.

 $\rm (NH_4)_2\,SO_4$ and a saturated solution at 20°C of ferrous sulfate heptahydrate ($\rm FeSO_4\bullet7H_2O).$ Ethanol was obtained from the distillation of sugar cane juice at a 90% concentration which was then diluted to 15%.

 Table 1 Delignification conditions for each treatment and

agro-industrial waste

2. Experimental procedure

2.1 Sampling of agro-industrial waste

The AIW used in this study was obtained from the processing of sugar cane, cassava, rice, and banana. The waste samples were collected from the main agro-industrial companies located in the provinces of Coronel Portillo and Padre Abad in the Ucayali region of Peru. The banana and cassava peels were collected from the Campo Verde district, the sugarcane bagasse from the Neshuya district, and the rice husks from the Callería district (Figure 1).



Figure 1 Location of the sampling area

During the month of October 2020, a total of 15 kg of AIW comprising sugarcane bagasse, banana peels, cassava peels, and rice husks were collected (Figure 2). The sugarcane bagasse was obtained from companies involved in producing alcohol by fermentation of sugarcane; the banana peels were obtained from *chifles* (thin slices of fried green banana) production; the cassava peels were obtained from cassava starch production; and the rice was obtained from a company dedicated to rice milling.

2.2 Delignification treatments

Four delignification treatments were compared to obtain cellulose pulp from the AIW under study: 10% sodium hydroxide, 50% ethanol, distilled water, and 25% Mohr's salt. The mass (g) to volume (ml) ratio and cooking time for each treatment are '©ven in Table 1. Each treatment was performed in triplicate.

Analytical-grade reagents were used in this research work. The 10% sodium hydroxide solution was prepared with 88.5% sodium hydroxide. Mohr's salt was prepared with a 40% saturated solution of ammonium sulfate

Treatment	Waste (g:ml		Time (min)	
	Banana	1:10		
	peel			
	Cassava	1:10		
10% sodium	peel		75	
hydroxide	Cane	1.1:15		
	bagasse			
	Rice husk	1.1:15		
	Banana	1:18.5		
	peel			
	Cassava	1:18.5		
Distilled	peel		60	
water	Cane	1.1:15		
	bagasse			
	Rice husk	1.1:15		
	Banana	1:8		
	peel			
	Cassava	1:8		
50%	peel		60	
ethanol	Cane	1.1:15		
	bagasse			
	Rice husk	1.1:15		
	Banana	1:8		
	peel			
	Cassava	1:8		
25% Mohr's	peel		75	
salt	Cane	1.1:15		
	bagasse			
	Rice husk	1.1:15		

2.3 Extraction of cellulosic pulp

The methodology used for the delignification of the residues and cellulose extraction is illustrated in Figure 3. As a pre-treatment, the residues were chopped with scissors and then ground with a hand mill. Once each residue was weighed, it was subjected to its respective treatment. Cooking was carried out in a semi-industrial three-burner gas stove at a constant temperature, for the time established for each treatment. During this process, the temperature was measured using a digital laser infrared thermometer. Washing was done with pressurized well water and the volume of water used was related to the reagent release from the pulp. The cleaned sample was filtered with a stainless-steel strainer and the resulting pulp was oven-dried at 105 °C for 24 hours.

2.4 Cellulose, hemicellulose, and lignin analysis

Cellulose, hemicellulose, and lignin content were determined before and after treatments. Prior to cellulose,



Figure 2 Samples of banana peel (a), cassava peel (b), sugarcane bagasse (c), and rice husks (d)



Figure 3 Delignification process of the AIW evaluated

hemicellulose, and lignin analysis; extracts were removed from the sample. Extractives are compounds soluble in neutral solvents that can interfere with subsequent chemical analysis. The TAPPI 204 (Technical Association of Pulp and Paper Industry – Extractive-free sample) norm was followed.

Holocellulose content was estimated by the ASTM (American Society for Testing and Materials) D-1104 method. 2 g of extract-free sample was placed into a 250 ml bottle containing 150 ml of distilled water, 0.2 ml of acetic acid, and 1 g of sodium chlorite. The bottle was placed in a water bath at 70-80°C, and every hour, for 5 hours, 0.22 ml of very cold acetic acid and 1 g of sodium chlorite was placed. After five hours, the bottle was placed

in an ice bath until it reached 10°C. Finally, we filtered the sample and washed it with 500 ml of cold water. The sample was dried at 105°C until a constant weight was obtained.

After this procedure, the cellulose content was determined according to the ASTM 1695-77 norm. For this test, 2 g of holocellulose were sampled and mixed with 10 ml of 17.5% sodium hydroxide at a constant temperature of 20°C in a thermoregulating bath. After 2 minutes, 5 ml of sodium hydroxide solution were added at 5-minute intervals until a total of 25 ml was added. The mixture was then stirred at 20°C for 45 minutes. We added 33ml of distilled water at 20°C, and the crucibles with the samples were allowed to stand for one hour before washing them with distilled water. Then we added 15 ml of 10% acetic acid to the cellulose collected in the crucible. After that, the acid was removed, leaving the sample slightly covered for 3 min. Finally, the sample was dried to determine the weight of cellulose.

The percentage of acid-insoluble lignin was determined with the sample free of extractives, using the procedure of the TAPPI T222 om-98 standard or Klasson method. We weighed 1 g of extractive-free sample and placed it in a 50 ml beaker. Then we added 15ml of 72% sulfuric acid and stirred it for 2 hours until the sample acquired a blackish color. The sample was then transferred to a 1L beaker, and the sulfuric acid was diluted to 4% by adding distilled water, and gently boiled for 4 hours. After that time, the sample was decanted, filtered, and dried at 105 °C for 24 hours.

After each treatment, the pulp yield was obtained with Equation 1 [20].

$$\text{Yield} \ (\%) = \frac{P_S}{M_S} x 100 \tag{1}$$

Where Ps the dried weight of the pulp after treatment and Ms is the initial dried weight of the pulp.

2.5 Statistical analysis

Statistical analysis was performed using SPSS 26 software. The difference between treatments was analyzed using a one-way analysis of variance (Anova) and Tukey's test at a confidence level of 95%. Pearson's correlation coefficient was used to determine the relationship between temperature and yield.

3. Results and discussion

3.1 Evaluation of chemical composition

Table 2 presents the chemical composition of the AIW under study. Our results indicate that this residue has the greatest potential for cellulose utilization compared to other waste materials. Specifically, we found that cassava peels have the lowest proportion of cellulose and hemicellulose among the residues studied. In contrast, the cellulose, hemicellulose, and lignin contents of banana peel vary greatly from those reported in other studies. Many authors have attributed these differences to variations in climate, geography, crop type [21], and species [22, 23], highlighting the of conducting thorough chemical analyses when evaluating potential sources of cellulose fibers.

Furthermore, our study revealed that rice husks have a higher initial cellulose content than the other residues studied. Table 2 provides detailed information about the cellulose, hemicellulose, and lignin contents of the AIW under study, supporting our conclusions about the relative cellulose content of the different residues. Our results provide valuable insights into the chemical composition of these Amazonian AIW and highlight the potential for their use in cellulose production.

3.2 Efficiency of treatments for waste delignification

Figure 4 shows the initial and final content of cellulose (Figure 4a), hemicellulose (Figure 4b), and lignin (Figure

Waste/Reference	Cellulose	RHemicellulose	Lignin
Banana peel			
This study	22.9	21.7	28.8
[24]	0.47	35.25	14.94
[25]	7.5	74.9	7.9
Rice husk			
This study	29.9	13.5	22
[26]	32.7	21.3	15.3
[27]	25-35	18-21	26-31
Cassava peel			
This study	19.3	8.1	25.2
[28]	14.8	50.3*	12.8
Sugarcane bagasse			
This study	24.2	20.2	25
[29]	44.43	22.9	17.52
[30]	45-50	25-30	2.4-9
[31]	35.61	32.29	22.56

 Table 2 Delignification conditions for each treatment and agro-industrial waste

*: Polysaccharides (mainly starch + hemicellulose)

4c) for each of the four AIW treatments evaluated. Sodium hydroxide was the most effective treatment for hemicellulose removal in banana peel and rice husks. Mohr's salt was the most effective treatment for hemicellulose removal in sugarcane bagasse. In the case of cassava peels, no treatment was able to reduce the hemicellulose content. Sodium hydroxide was the most effective treatment for the removal of lignin in banana peels, rice husk and sugarcane bagasse. The four treatments achieved similar final lignin concentrations in cassava peels (4.1 to 6.5%), with Mohr's salt resulting in the lowest concentration. Cassava had a very low final cellulose content with all treatments (between 18% and 23.5%).

Banana peel and rice husk showed a greater increase in cellulose concentration with the sodium hydroxide treatment. Herlina et al. [32] investigated the effects of sodium hydroxide on the chemical composition of corn husks. They reported that sodium hydroxide treatment was effective in removing non-cellulosic compounds, such as lignin, and increasing the cellulose content in both materials. For sugarcane bagasse, all treatments had similar final cellulose concentrations (39.9 to 48.1%). with the ethanol treatment resulting in the highest concentration. Bernier et al. [33] compared the efficiency of different pretreatment methods, including ethanol, on sugarcane bagasse. They found that all treatments resulted in similar final cellulose concentrations, but the ethanol treatment had the highest yield. In the case of cassava peels, the Mohr's salt treatment resulted in the highest final cellulose concentration (4.2%), although the increase was very limited.

For banana peel, rice husk, and sugarcane bagasse, the treatment with sodium hydroxide was able to considerably

Treatment	Waste	Time (min)	Temperature (°C)	Yield (%)	Pearson correlation coefficient	p-value	R2 (%)
10% sodium hydroxide	Banana peel	75	77.9	72.4	0.439	0.71 1	19 %
	Rice husk	75	77.3	65.3	-0.857	0.34 4	73 %
	Cane bagasse	75	72.6	38.5	0.11	0.99 3	1%
	Cassava peel	75	74.7	20.4	0.990	0.09 1	98 %
Distilled water	Banana peel	60	55.3	71.3	-0.615	0.57 8	38 %
	Rice husk	60	58.8	77.6	-0.809	0.40 0	65 %
	Cane bagasse	60	48.3	51.2	-0.500	0.66 7	25 %
	Cassava peel	60	56.3	18.3	-0.819	0.38 9	67 %
50 % ethanol	Banana peel	60	54.2	76.8	-0.115	0.92 7	1%
	Rice husk	60	47.3	64.6	-0.996	0.05 6	99 %
	Cane bagasse	60	0.50	50.4	-0.945	021 7	89 %
	Cassava peel	60	52.4	18.0	0.260	0.83 3	7%
25% Mohr's salt	Banana peel	75	58.2	38.0	-0.884	031 0	78 %
	Rice husk	75	52.6	60.9	0.504	0.66 3	25 %
	Cane bagasse	75	50.5	48.9	-0.171	0.89 1	3%
	Cassava peel	75	48.4	23.4	0.091	0.94 2	1%

Table 3 Relation between temperature and yield

Note.**. Correlation is significant at the 0.01 level (bilateral). *. Correlation is significant at the 0.05 level (bilateral).

increase the cellulose composition by effectively removing non-cellulosic compounds, especially lignin [34]. The same occurred in cassava peel with Mohr's salt treatment, although the increase in the proportion of cellulose was very limited. Herlina et al. [32] investigated the effect of sodium hydroxide on the chemical composition of corn husks. The study reported that the treatment was effective in removing non-cellulosic compounds and increasing the cellulose content. Banana peel had the highest yield among the other residues; despite having a higher lignin content, which could be detrimental to the amount of time, inputs, and energy required to obtain cellulose pulp [35]. Cabascango et al. [36] investigated the yield and quality of cellulose pulp obtained from different agroindustrial residues, including banana peels. They reported that banana peels had the highest yield of cellulose pulp despite having a higher lignin content, which required more time, inputs, and energy for removal.

The main advantages of sodium hydroxide treatment are lignin removal and increased cellulose availability. Sodium hydroxide treatment causes biomass swelling, which increases the internal surface area of lignocellulosic particles and weakens the structural integrity of lignocellulose, breaking the bonds between lignin and other carbohydrates such as cellulose and hemicellulose [31].

Organosolv treatments are usually done at temperatures between 160 and 220°C [35]. As the ethanol treatments were done at a relatively low temperature (between 47 and 54°C); it may have not been as effective for lignocellulosic degradation [37]. However, treatment conditions at higher temperatures are more expensive and require equipment designed with special materials to avoid corrosion processes [38].

Although hot water treatments are usually performed



(a)





Figure 4 Final cellulose (a), hemicellulose (b), and lignin (c) contents for each waste after treatment

at temperatures higher than 100 °C [39], the distilled water treatment had acceptable results in lignin removal and cellulose recovery. In hot water treatments, the lignocellulosic structure undergoes morphological and chemical changes, such as the deacetylation of hemicellulose and the rearrangement of the lignin structure [40]. It is possible that the same mechanism took place in this case.

Overall, under the evaluated conditions Mohr's salt had the greatest lignin removal rate. Surprisingly, the distilled water treatment was more effective than the alkaline treatment for lignin removal on banana peel, cassava peel, and risk husk.

The variability of the results highlights that the effectiveness of the treatments depends mainly on

the chemical and structural complexity of the polymers present in the lignocellulose, which vary with the origin and type of material [26]. These results lead to the conclusion that there is no single pretreatment technology applicable to different types of biomasses.

3.3 Relation between temperature and cellulose yield

One important factor to consider when evaluating the effect of temperature on cellulose yield is the specific treatment being used. In this study, four different treatments were evaluated, each with a different temperature. The average temperature for the treatment using sodium hydroxide, distilled water, ethanol and Mohr's salt were 75.6°C, 54.6°C, 50.9°C and 52.4°C, respectively. Although there is no significant correlation between temperature and yield (Table 3), a proportional relationship between temperature and yield was observed for each treatment. For example, the highest yield for banana peel was obtained with sodium hydroxide (72.4%), while the highest yield for rice husk was obtained with distilled water at 58.8°C (77.6%).

[37] studied the effect of substrate. Gabhane et al. reaction time, and temperature on the delignification and found that the most significant factor was the substrate. They observed that time and temperature had a negative and positive (although limited) relation, respectively, with sugar reduction yield. Correia et al. [41] reported a decrease in the reaction efficiency with increasing temperature (between 150 and 190 °C) and time (between one to three hours): however, the temperatures and times evaluated were higher than those in this study. These researchers indicated that lignin and hemicellulose removal is more effective at higher temperatures and shorter times. According to de Groot et al. [42], delignification occurs in three stages. In the first stage, a large amount of hemicellulose and little lignin is removed. In the second stage (the dominant one), there is a greater removal of lignin and hemicellulose. In the third stage, lignin removal occurs more slowly as a result of the greater inertness of the residual lignin in this residual phase. Therefore, it is not recommended that these treatments be applied at very high temperatures and for long periods of time.

These findings suggest that the relationship between temperature and cellulose yield can be complex and dependent on a variety of factors, including the specific treatment being used and the nature of the biomass being processed.

4. Conclusions

Agro-industrial activity in the Amazon region generates a constant and increasing amount of AIW. In this study, we evaluated four delignification treatments for cellulose recovery from AIWs from the Ucayali region in Peru. Among the four wastes evaluated, rice husks showed the highest potential for cellulose recovery, followed by banana peels, and sugarcane bagasse, while cassava peels had the lowest potential for cellulose recovery. The treatment with 10% sodium hydroxide showed the highest percentage of lignin removal in banana peels, rice husk and sugarcane bagasse. The treatment with Mohr's salt had the lowest yield for all wastes, while the treatment with distilled water at medium temperature (between 48 and 58°C) reduced the proportion of lignin and increased the proportion of cellulose similarly to conventional treatments (sodium hydroxide and ethanol). Interestingly, the treatment with distilled water showed potential as a simple, economical, and eco-friendly technology for delignification and cellulose recovery. Further characterization of the biomass after treatment with distilled water is needed to identify the predominant mechanisms involved. Overall, our findings demonstrate the great potential of AIWs as a source of cellulose and provide insights into effective and sustainable methods for their recovery.

5. Declaration of competing interest

We declare that we have no significant competing interests including financial or non-financial, professional, or personal interests interfering with the full and objective presentation of the work described in this manuscript.

6. Funding

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7. Author contributions

Grober Panduro Pisco: Conceptualization, Methodology, Writing the Original draft, , Fund acquisition. Lady Di Hoyos Shica: Formal analysis, Investigation. Edwar Edinson Rubina Arana: Methodology, Formal Analysis, Investigation. David Leon Moreno: Investigation.

8. Data availability statement

The datasets and data collection methods generated during and/or analyzed during the current study are

available from the corresponding author on reasonable request.

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