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Research Article

Characterization of Vietnamese Lacquer Collected in Different Seasons

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Vietnamese lacquers collected every month from June to March of next year were characterized. Composition analysis showed that lacquer collected in rainy season contained much water, while those collected in dry season contained more lipid component. Although hardness of lacquer films is not very hard, lacquers tapped in all seasons can reach hard dry (HD) within 48 hours. Refining lacquer can accelerate drying time but the water concentration should be maintained around 10 wt% for laccase activity.

1. Introduction

Lacquer is sap collected from lacquer tree, which belongs to Anacardiaceae and *Toxicodendron* families growing in Asia. It has been used as coating material for thousands of years in Asian countries due to durability and beauty [1–4]. Lacquer from *Toxicodendron vernicifluum* lacquer tree, which grows in China, Japan, and Korea, is usually collected from June to October, and the characters are somewhat different according to the collection month [5]. For example, lacquer tapped from June to July (rainy season in East Asia) contains much water and dries faster than another season. Lacquer tapped in August has better gloss and transparency due to the higher lipid component compared with other seasons. These characters of lacquer depend on the climate during the collection season. Therefore, in lacquer market, the price differs according to collection season and lacquer collected in August with high lipid content fetches the highest price.

In South Asia, especially in Vietnam, lacquer is collected from *Toxicodendron succedaneum* lacquer trees from January to December because of tropical climate [6]. Vietnamese people have used *T. succedaneum* lacquer sap as adhesive and coating material in their long lacquer culture history [7–11]. Composition and characterization of laccol [12] and synthesis of epoxy resin derivative from laccol [13] also have been reported. However, how to improve the quality of Vietnamese lacquer is rarely reported.

In the lacquer market, Vietnamese lacquer has relatively poor reputation because of its slow drying and soft film. It has been said that Vietnamese lacquer collected throughout the year is combined in one barrel. This practice may be one reason it has a reputation for low quality in international lacquer market. In order to better understand the real quality and characteristics of Vietnamese lacquer, Vietnamese lacquer saps collected every month from June of 2012 to March of 2013 were analyzed. The composition, drying time, laccase activity, and infrared absorption of lacquer film collected during those months were determined; the characteristics of Vietnamese lacquer are discussed. In addition, development of fast drying lacquer was also carried out by refining (*kurome*) process, and the results are also discussed.

2. Experimental

2.1. Materials. Lacquer sap was tapped from *Toxicodendron succedanea* lacquer tree growing in Tho Van (T6–T3 series) and Di Nau (D6–D3 series) communes of Tam Nong, Pho Tho, Vietnam, every month from June 2012 to March 2013. Because the quantity collected was inadequate for use in the *kurome* study, lacquer sap collected in September 2012 in Tho Van commune which was provided by United Nations Industrial Development Organization (UNIDO) of Vietnam was used in the *kurome* process.

2.2. Method. Lacquer viscosity was determined at room temperature using a Brookfield Engineering Laboratories programmable DV-II viscometer with a CPE-51 spindle. The rotation speed was 5–20 rpm, and the measurement volume was 0.5 mL.

Drying time was carried out using an automatic drying time recorder (Tai Yu Equipment, Osaka, Japan), and the thickness of the tested lacquer films on the glass plate was 76 μm when wet as we previously reported [14].

The hardness and color change of lacquer film were measured by pencil hardness tester (Yoshimituseiki, Japan) and Color-Guide instrument (BYK-Gardner, Germany), respectively. ATR-IR spectra of lacquer films were measured using JASCO FT-IR410 spectrophotometer.

Laccase activity was determined using 0.216 mmol/L syringaldazine/methanol as substrate in 0.1 mmol/L sodium phosphate buffer (pH 6.5) and measured by monitoring the increase in UV absorbance at 525 nm at 30°C. One unit of enzyme was equal to an increase of 0.001 absorbance unit per min per mg of acetone powder (AP).

The *kurome* process is microdispersal of water molecules and dehydration of lacquer sap by mixing. In this study, the *kurome* process was carried out by changing rotating speed and mixing time, as described in our previous report [15].

Molecular weight distribution and average molecular weight were determined at 40°C by gel permeation chromatography (GPC, TSK-gel columns α-3000, α-4000, and α-M, φ7.8 mm × 300 mm × 3, Tosoh). Dimethylformamide (DMF) with 0.01 mol of LiBr was used as an eluent on a high-performance liquid chromatography system (HPLC). A refractive-index detector was used and polystyrene having molecular weights of 1.90×10^5, 4.39×10^4, 5.40×10^3, and 5.00×10^2 g/mol was used as standards. The elution rate and pressure of the DMF eluent were 0.8 mL/min and 48 kg/cm^2, respectively.

3. Result and Discussion

3.1. Composition Analysis of Season Lacquers. After measuring the viscosities, lacquer samples were separated with acetone to obtain lipid component and acetone powder (AP). The separation results and viscosities are summarized in Table 1.

Table 1: Compositions of season Vietnamese lacquer.

Samples	Area	Month	Lipid (%)	AP (%)	Water (%)	Viscosity (mPas)
T6	Tho Van	6	43.22	21.38	35.39	4447
T7		7	43.85	21.23	34.92	3113
T8		8	62.40	14.17	23.43	549
T9		9	55.56	15.74	28.70	1088
T10		10	61.32	13.11	25.57	726
T11		11	62.17	13.15	24.68	778
T12		12	59.35	13.91	26.74	713
T1		1	62.26	13.58	24.16	818
T2		2	29.43	24.71	45.86	8865
T3		3	29.84	24.22	45.94	7525
D6	Di Nau	6	70.42	11.12	18.47	1012
D7		7	39.23	23.43	37.34	3659
D8		8	37.42	25.20	37.38	7387
D9		9	46.49	20.21	33.30	1779
D10		10	61.34	13.84	24.82	811
D11		11	66.13	13.85	20.02	713
D12		12	38.87	24.49	36.64	2195
D1		1	59.03	16.87	24.10	726
D2		2	49.73	20.30	29.97	1300
D3		3	44.52	21.37	34.10	1884

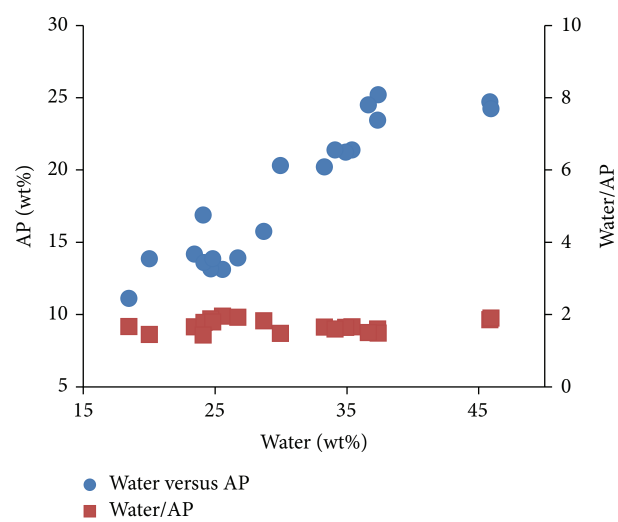

Figure 1: Ratio of water to AP.

The lacquers collected from June to September (T6–T9, D6–D9) had higher water concentration compared with those collected from October to January (T10–T1, D10–D1). May to October is rainy and November to March is dry season in Vietnam. Obviously, the water concentration in lacquer is related to climate, and this phenomenon affected lacquer collected from *Toxicodendron vernicifluum* lacquer tree growing in China, Japan, and Korea. Although February and March are not a rainy season, lacquer collected in both months had a high water concentration, which can be considered due to high humidity in this season in Vietnam. However, we found that the ratio of AP increased with increasing concentration of water, and the water concentration to AP ratio was constant as shown in Figure 1. This phenomenon means that the higher water concentration in lacquer sap is not simply matter of rain or humidity when collected in rainy season or the very humid months of February and March. A lacquer tree is a higher plant, its life activity is very complex, and there is much that we humans do not understand. It can also be seen that seasonal lacquer viscosities increased with increasing water concentration, as shown in Figure 2. That is, the energy of emulsification is increased with increasing percentage of water in lipid.

The drying time and hardness of lacquer films are summarized in Table 2. The drying time does not differ much by collecting season, because the drying time is related to

Table 2: Compositions of season Vietnamese lacquer.

Samples	Drying time[1] (h)			Hardness[2] for drying days								
	DF	TF	HD	1	2	3	4	5	6	7	14	21
T6	4.3	5.4	8.0	<6B	<6B	<6B	<6B	<6B	<6B	<6B	<6B	4B
T7	3.8	4.8	8.8	<6B	<6B	<6B	<6B	<6B	<6B	<6B	<6B	5B
T8	2.6	3.2	6.0	<6B	<6B	<6B	<6B	<6B	<6B	<6B	<6B	4B
T9	6.5	7.5	15.0	<6B	<6B	<6B	<6B	<6B	<6B	<6B	5B	5B
T10	2.8	3.5	6.5	<6B	<6B	5B	5B	5B	5B	5B	3B	3B
T11	4.0	5.0	8.5	<6B	<6B	<6B	6B	5B	5B	5B	3B	3B
T12	4.0	4.7	8.5	<6B	<6B	<6B	6B	5B	5B	5B	3B	3B
T1	7.8	8.5	16.0	<6B	<6B	<6B	<6B	<6B	<6B	<6B	4B	4B
T2	2.8	3.5	6.0	<6B	<6B	<6B	<6B	<6B	<6B	4B	4B	4B
T3	2.0	3.0	4.5	<6B	<6B	<6B	<6B	<6B	<6B	4B	4B	3B
D6	14.0	15.5	<48	—	<6B	<6B	<6B	<6B	<6B	<6B	<6B	<6B
D7	6.0	8.2	<48	—	<6B	<6B	<6B	<6B	<6B	<6B	<6B	4B
D8	3.8	5.5	<48	—	<6B	<6B	<6B	<6B	<6B	<6B	<6B	<6B
D9	2.7	3.2	4.5	<6B	<6B	6B	5B	5B	5B	5B	3B	3B
D10	2.5	3.0	5.0	<6B	<6B	<6B	5B	5B	5B	5B	4B	4B
D11	6.7	7.0	15.0	<6B	<6B	<6B	5B	5B	5B	5B	4B	4B
D12	3.0	4.0	5.5	<6B	<6B	<6B	<6B	<6B	<6B	<6B	4B	4B
D1	3.8	4.3	8.0	<6B	<6B	<6B	<6B	<6B	<6B	<6B	4B	4B
D2	1.5	2.0	3.5	<6B	<6B	<6B	<6B	<6B	<6B	<6B	4B	3B
D3	1.5	2.0	3.0	<6B	<6B	<6B	<6B	<6B	<6B	4B	4B	3B

[1]Drying condition: 25°C, 80%RH, and wet thickness 76 μm. DF: dust-free dry; TF: touch-free dry; HD: hard dry.
[2]Based on pencil hardness test method.

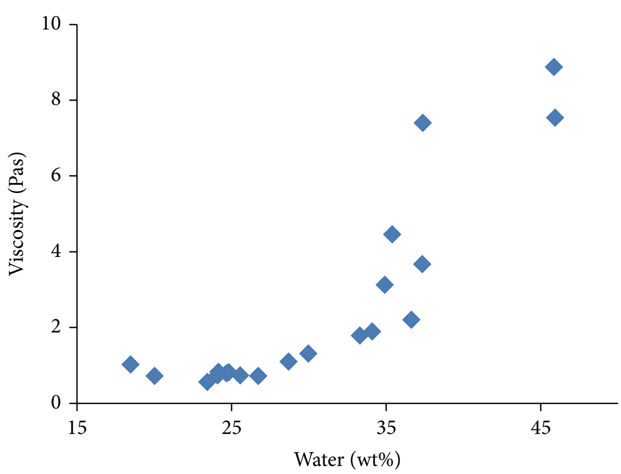

Figure 2: Viscosity of lacquer sap.

laccase concentration and activity, and almost all collecting season samples have similar laccase concentration and activity (Table 3). On the other hand, the film hardness is relative to the percentage of lipids in the lacquer sap. Hardness is strongly influenced by the lipid concentration because autoxidation of lipid side chain causes film hardness.

The laccase activity contained in AP was measured using syringaldazine as substrate, and the results are summarized in Table 3. Like drying time, no significant difference was found in the laccase activity by collecting season, and this results in almost the same drying time (Table 2).

Table 3: Lacquer laccase activities.

Samples	Activities (unit/mg AP)	Samples	Activities (unit/mg AP)
T6	555	D6	693
T7	556	D7	416
T8	750	D8	538
T9	552	D9	455
T10	669	D10	710
T11	645	D11	528
T12	697	D12	329
T1	614	D1	746
T2	570	D2	534
T3	617	D3	447

Table 4: Composition percentage, lipid/AP, and contact angle of lacquer films.

Samples	Composition percentage (wt%)			Lipid/AP	Contact angle (degree)
	Lipid	AP	Water		
T3	29.8	24.2	45.9	1.23	67.0
T7	43.8	21.2	34.9	2.07	72.5
T11	62.2	13.2	24.7	4.73	77.0

Because components are rather different depending on the collection season and lacquer dries by evaporation of water, we hypothesized that dry lacquer films have different properties. In order to reveal the reason, we analyzed the contact angle of drying lacquer films, T3, T7, and T11, and the results are summarized in Table 4. It can be seen that

Table 5: Data results of kneaded lacquers.

Kneading speed (rpm)	Kneading time (min)	Water (%)	Viscosities (mPas)	MW distribution (%)[1]			Drying time[2] (h)		
				Monomer	Oligomer	Polymer	DF	TF	HD
—	—	35.4	1203	86.8	13.0	0.2	7.8	8.1	12.5
60	15	26.4	1400	73.8	25.6	0.6	12.2	13.8	31.5
60	30	20.4	2442	56.5	41.8	1.7	6.6	7.2	11.8
60	45	13.6	2694	50.1	48.1	1.8	2.8	3.3	5.5
60	60	12.5	4639	44.8	52.8	2.4	2.2	2.7	5.2
120	15	27.1	3192	66.9	32.6	0.5	15.8	19.0	96
120	30	16.6	2747	54.1	45.0	1.0	3.8	4.5	7.5
120	45	8.1	3558	51.3	45.3	1.6	3.0	3.4	4.5
120	60	5.2	3625	50.2	47.4	2.4	3.4	3.8	6.0
180	15	21.5	1936	67.4	32.0	0.6	16.2	18.5	72
180	30	9.0	2250	56.1	42.7	1.1	1.7	2.8	6.0
180	45	5.2	2389	55.7	42.7	1.6	3.3	3.6	7.0
180	60	4.3	3322	52.3	45.8	1.9	3.1	3.7	6.0

[1]Determined by GPC. Monomer: MW = 320; oligomer: 10000 > MW > 600; polymer: MW > 10000.
[2]Drying condition: 25°C, 80%RH, and wet thickness 76 μm. DF: dust-free dry; TF: touch-free dry; HD: hard dry.

Table 6: Lightness, gloss, and coloration of kneaded lacquer films[1].

Kneading speed (rpm)	Kneading time (min)	Lightness and coloration[2]			Gloss
		L^*	a^*	b^*	
—	—	22.66	33.27	22.78	8.9
60	15	31.42	40.09	49.75	72.8
60	30	20.57	38.84	33.33	76.6
60	45	9.40	27.52	12.50	85.8
60	60	6.08	25.27	9.05	98.2
120	15	35.87	37.43	58.74	98.3
120	30	11.78	30.72	16.53	92.2
120	45	6.22	25.32	9.26	96.1
120	60	5.03	21.42	7.18	96.6
180	15	40.24	37.22	65.49	99.0
180	30	6.32	25.99	9.57	96.7
180	45	5.05	21.21	7.16	97.0
180	60	6.47	21.91	7.23	95.2

[1]Drying condition: 25°C, 80%RH, and wet thickness 76 μm.
[2]L^*: lightness; a^*: +red/−green; b^*: +yellow/−blue.

the contact angle increased with decreasing percentage of AP. Figure 3 shows ATR-IR spectra of T3, T7, and T11 lacquer films. Increasing stretch vibrations of hydroxyl group (O–H) at 3330 cm^{-1} and ether (C–O) or alcohol (C–OH) at 1037 cm^{-1} were discernible with increasing percentage of AP. The major component of AP is polysaccharide, and polysaccharides dissolve in water micelles that are dispersed in the W/O emulsion of lacquer sap to form a film after drying, when the polysaccharide usually appears at the surface of lacquer film. It was suggested that increasing polysaccharides made lacquer film more hydrophilic.

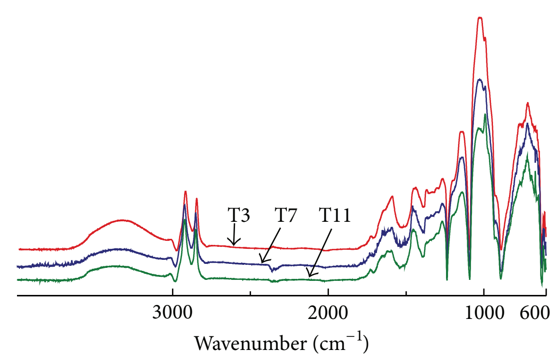

Figure 3: ATR-IR spectra of lacquer film.

From the above analyses, it can be concluded that although selecting the best season to collect high quality Vietnamese lacquer sap is difficult, sap collected in dry season (from September to December) has a higher percentage of lipid that gives the film high hardness, and the sap collected in other seasons has a higher percentage of water and AP that makes the film more hydrophilic.

3.2. Development of Fast Drying Lacquer. In order to improve the performance of Vietnamese lacquer, we performed differential *kurome* process with kneading speed of 60, 120, and 180 rpm and kneading time of 15, 30, 45, and 60 min, as summarized in Table 5. During the *kurome* process, the water concentration decreased with increasing kneading speed, and enzymatic polymerization of the lacquer sap was promoted. The polymerization of lacquer sap is catalyzed by the laccase contained in water micelles and requires oxygen. A high kneading speed beats in more oxygen and promotes polymerization. On the other hand, a high kneading speed also quickly evaporates water, which then decreases laccase

Table 7: Data results of repeated *kurome* lacquers.

Water addition	Kneading time (min)	Water (%)	Viscosities (Pas)	MW distribution (%)[1]			Drying time[2] (h)		
				Monomer	Oligomer	Polymer	DF	TF	HD
None	0	33.6	1.2	86.8	13.0	0.2	7.8	8.1	12.5
	60	5.2	6.6	50.2	47.5	2.3	3.0	3.6	8.3
	90	4.3	9.0	45.9	51.2	2.8	4.4	7.4	8.6
	120	4.1	10.8	45.3	51.7	3.0	4.4	5.3	8.6
	180	4.4	10.6	45.0	51.6	3.3			
5%/30 min	60	7.1	6.1	49.3	48.0	2.7	2.9	3.3	8.3
	90	4.5	7.8	44.6	52.4	3.1	3.4	3.8	5.3
	120	5.1	8.4	43.5	53.2	3.3	3.5	3.9	7.7
	180	3.7	12.6	39.5	56.3	4.1			
10%/30 min	60	7.8	4.2	56.1	41.2	2.7	3.4	4.2	8.6
	90	5.9	6.3	47.1	49.3	3.6	3.8	4.3	8.3
	120	4.1	10.9	40.6	54.6	4.7	3.3	3.6	7.5
	180	3.8	22.4	35.9	58.2	5.9			
20%/30 min	60	7.3	5.8	48.2	47.5	4.2	3.2	4.5	8.3
	90	5.5	11.2	39.6	54.8	5.6	5.5	6.4	9.0
	120	9.5	23.4	33.1	58.2	8.7	2.6	3.4	6.0
	150	12.3	43.5	30.5	59.4	10.1			

[1]Determined by GPC. Monomer: MW = 320; oligomer: 10000 > MW > 600; polymer: MW > 10000.
[2]Drying condition: 25°C, 80%RH, and wet thickness 76 μm. DF: dust-free dry; TF: touch-free dry; HD: hard dry.

activity. In our previous work [16, 17], it was found that lacquer sap collected from *T. vernicifluum* lacquer trees can be polymerized to form a film even if the water concentration is just over 3 wt%. However, in this study, we found that the lacquer sap collected from *T. succedanea* lacquer trees was not polymerized when the water concentration was lower than 10 wt%. Because the AP concentration in *T. succedanea* lacquer sap is higher than that in *T. vernicifluum* lacquer sap, this difference suggested that *T. succedanea* sap requires more water to dissolve the laccase and become polymerized. In general, viscosity of lacquer sap decreases with increasing water concentration. Therefore, the viscosity of kneaded lacquer increased with progression of polymerization due to decrease of water concentration during the kneading process, and the drying time of kneaded lacquer was also substantially shortened with the progress of polymerization.

Gloss, L^*, a^*, and b^* are summarized in Table 6. Gloss reached 100 for lacquer saps kneaded for 15 min at 120 and 180 rpm. Kneading at a speed of 60 rpm which needed 60 min made gloss reach 100. Because water micelles contained in lacquer sap can be microdispersed by kneading and stirring, the film surface becomes smooth and the gloss increases. Water micelles can be miniaturized quickly when the kneading speed is high. At all kneading speeds, L^*, a^*, and b^* all decrease as the kneading time increased suggesting that the lacquer film become transparent and color change from red/yellow to green/blue.

In this study, we found that if the water concentration in Vietnamese lacquer is lower than 10 wt%, which was kneaded at 120 rpm for 45 and 60 min and 180 rpm for 30, 45, and 60 min (Table 5), the polymerization of laccol catalyzed by laccase should not occur. In order to prevent a decrease in the water concentration, repeated *kurome*, which is the addition of water every 30 min during the kneading, was carried out. The results are summarized in Table 7. *Kurome* can accelerate laccase polymerization of lacquer because as kneading time increases and is repeated, monomer decreases and oligomer increases, and polymer also slightly increases. This phenomenon is consistent with the results from *kurome* lacquer sap collected from *T. vernicifluum* lacquer tree [18].

4. Conclusions

Lacquer saps collected from *Toxicodendron succedanea* lacquer tree growing in Vietnam during the rainy season contained a relatively high concentration of water and during the dry season contained a relatively high concentration of lipid component. The compositions of Vietnamese lacquer are affected by collecting season like the lacquer sap collected from *Toxicodendron vernicifluum* lacquer tree growing in China, Japan, and Korea. The increasing ratio of AP makes the film hydrophilic, and the increasing ratio of lipids increases the film hardness. After being refined by the *kurome* process, the lacquer polymerizes faster than in its raw state. The polymerization stopped when the amount of water fell below 10 wt%, but the polymerization can be promoted further by adding water during the kneading process, and this is called the repeat-*kurome* process.

Conflict of Interests

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

Acknowledgments

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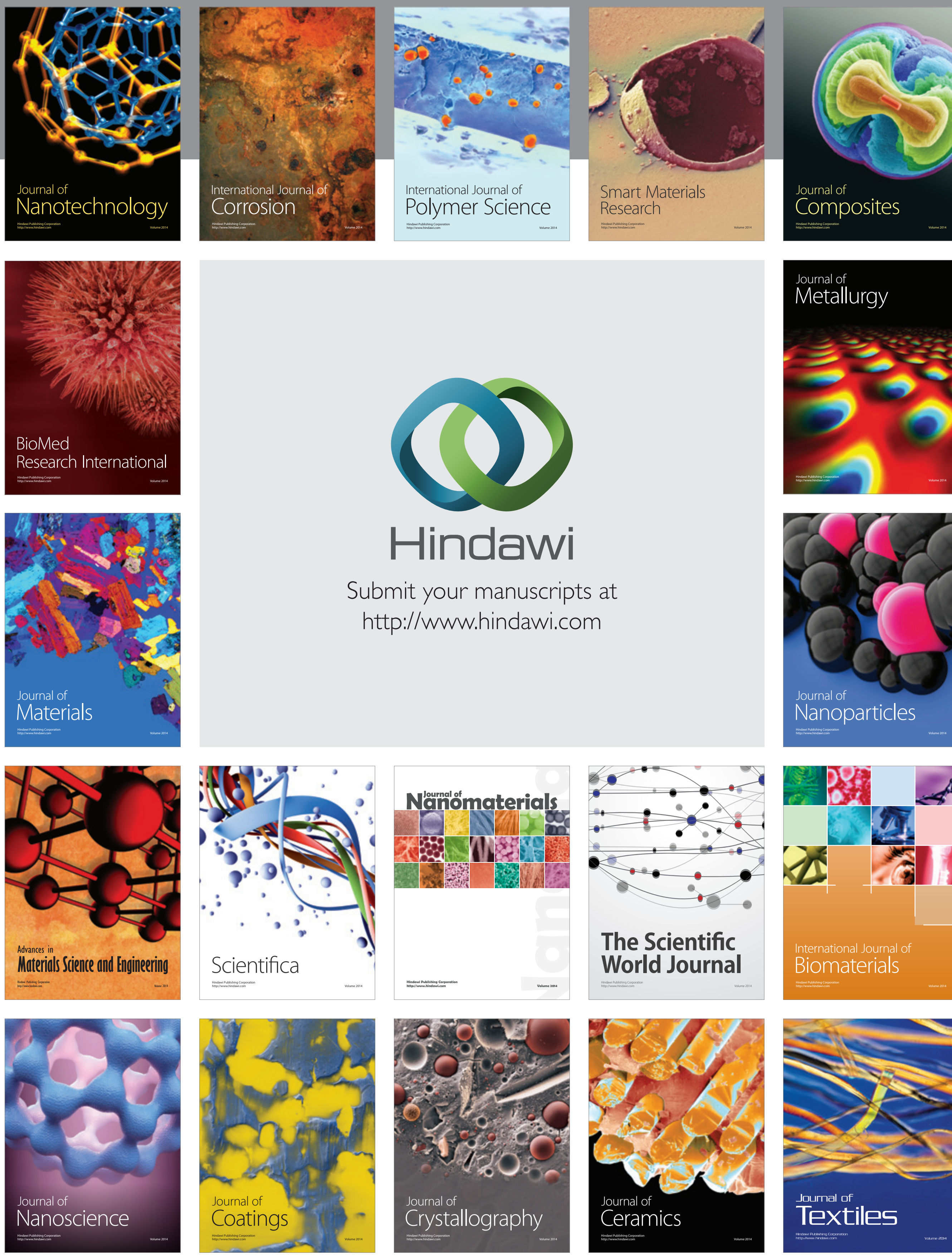
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