Recycling Wood Composite Panels: Characterizing Recycled Materials

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Downgraded medium density fiberboard (MDF), particleboard (PB), and oriented strandboard (OSB) panels were individually subjected to steam explosion treatment. Downgraded MDF and PB panels were separately treated with thermal chemical impregnation using 0.5% butanetetracarboxylic acid (BTCA). And downgraded PB panels were processed with mechanical hammermilling. The pH, buffer capacity, fiber length, and particle size of these recycled materials were evaluated. After the steam explosion and thermal chemical impregnation treatments, the pH and buffer capacity of recycled urea formaldehyde resin (UF)-bonded MDF and PB furnishes increased and the fiber length decreased. The hammermilling of recycled PB was less likely to break particles down into sizes less than 1 mm^2.

Keywords: Recycling; MDF; PB; OSB; Steam explosion; Impregnation thermal treatment; Hammermill; pH; Buffer capacity

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INTRODUCTION

In 2013, the consumption of particleboard (PB) and medium density fiberboard (MDF) in the US totaled 2.87 and 2.66 billion square feet, respectively. It is expected that in 2014 the consumption of PB and MDF panels will increase by 5.8% and 13.7%, respectively (RISI 2014). Given this information, it is obvious that every year a huge amount of wood composite panels will need to be disposed of or recycled after the completion of their service life.

To address the wood composite recycling issue, several methods have been developed for reconstituting PB and MDF wastes into new products, including chemo-thermo-mechanical processing (Michanickl 1996a,b; Boehme and Michanickl 1998; Roffael *et al.* 2010), hydrothermal treatment (Franke and Roffael 1998; Lykidis and Grigoriou 2008, 2011), chemical treatment (Michanickl 1997), and mechanical treatments (Ye *et al.* 1998; Roffael 2002; Roffael *et al.* 2002; Czarnecki *et al.* 2003). These research results have revealed that urea-formaldehyde (UF) and phenol-formaldehyde (PF) resins can be used to produce PB and MDF from recycled materials using conventional technologies designated for PB and MDF manufacturing. Although the research and technologies such as Fibresolve (Sandison 2002; Wastesave 2014) and C-Tech Innovation (WBPI 2014) have demonstrated the feasibility of the process, the wood composite industry in the USA and Canada is reluctant to use more than 15% of recycled or downgraded UF resin bonded panel materials in manufacturing, for example, PB panels.

Recycling used wood composite panels for their reconstitution into new panels is a complex process that involves the preparation of wood raw materials, selection of the right resin system, and optimization of the hot press strategy. Therefore, to produce wood composites made from recycled materials efficiently, an understanding of the physical and chemical properties of wood and recycled materials, the resin manufacture process, resin chemistry, the hot press process, and the interactions between wood, recycled material, resin, and panel manufacturing conditions is crucial.

In this current study, three different recycled composites (MDF, PB, and OSB) were treated with steam explosion, chemical impregnation together with thermal treatment, and hammermilling. The purpose of this study was to understand how these treatments individually affected the chemical and physical properties of the recycled materials examined. Potential strategies regarding the efficient use of recycled materials are discussed.

EXPERIMENTAL

Downgraded MDF, PB, and OSB panels were collected from MDF, PB, and OSB mills, respectively. Their characteristics are listed in Table 1 below and their features are described in the following discussion. They were kept dry at the research lab of FPInnovations, Quebec, under ambient conditions until being used for the experiments.

Table 1. Used Panel Characteristics

Panel type	Dimension (ft x ft x in.)	Density (lb/ft^3)	% Resin	%wax	Wood species
MDF	4x8x5/8	48	12%UF in face and 10% in core layers	1.5% Ewax in face and 1% in core layers	SPF
PB	4x8x5/8	43	10% in UF in face and 8% in core layers	1.5% Ewax in face and 1% in core layers	SPF
OSB	4x8x7/16	40			90% Aspen and 10% softwood
One can convert feet to meter with factor of 0.3048 One can convert inch to meter with factor of 0.0254 One can convert lb/ft^3 to kg/m^3 with factor of 16 UF resins were catalyzed with 1% of 20% NH_4Cl solution for face layers and 0.75% for core layers, based on liquid resin weight Ewax or emulsified wax with 58% solid content was applied together with UF resin. SPF stands for white spruce, jackpine, and balsam fir In the10% softwood, it may contain red pine, white pine, jack pine, balsam fir, white spruce etc.					

Steam Explosion of MDF, PB, and OSB Panels

A continuous steam explosion reactor (Brown and Bender 1980) was used to break chips down into fibers. The recycled materials, *i.e.*, MDF, PB, and OSB panels, were first cut into strips about 2 inches (5.08 cm) wide and then were chipped by a DIEHL type strip cutter. Any oversized materials were separated with a 1-mesh screen. The materials that passed through the screen were used for the steam explosion treatment.

The procedure of steam explosion of recycled wood composite materials can be described as follows:

1) A mechanical feeder (a feeder hopper) supplies recycled materials to a pressurized vessel with a reciprocating piston in such a manner that the pressure difference can be maintained on a continuous basis. The feeder hopper is equipped with a water sprinkler system that supplies water to the material prior to its entry into the reactor vessel. The stainless steel pressure vessel measures 0.6 m in diameter and about 7 m in length with a screw conveyor running at 34 rpm through its interior. In this experiment, a certain amount of tap water was added such that the final steam-exploded recycled materials would reach moisture content around 65%.
2) As the material is fed in the vessel, saturated steam at a maximum temperature of 235 °C is applied simultaneously to the vessel. The screw conveyor carries the materials through the reactor at a designated rate and mixes and shreds the materials into smaller sized particles.
3) Once the steam treatment time is reached, an electronic valve can be used to quickly open and close the pressurized vessel to allow the material to move to a material collection cyclone. The pressure difference between the vessel and the atmosphere creates an explosive effect and further breaks the materials down into fibers. The steam explosion treatment time and temperatures used in this experiment are presented in Table 2.

Table 2. Steam Explosion Treatment Time and Temperature

	OSB	MDF	PB
Time (min)	1.87	0.80	0.80
Temperature (°C)	225	227	227

Chemical Impregnation of MDF and PB Panels

This part of the experiment was performed to verify the patent of Boehme and Michanickl (1998) but with different chemicals. A typical process used in this study was as follows: samples of 1000 g of downgraded PB or the same amount of MDF were impregnated with the same amount of 0.5% butanetetracarboxylic acid (BTCA) and 0.5% sodium hypophosphite hydrate (NaH_2PO_2) water solution under a vacuum (700 mmHg) for 10 min and then autoclaved using a Medalist 200 at 113 °C for 20 min. So the retention of the solution of treated panels was 100%. A control experiment using only tap water was also conducted.

Hammermilling Recycled PB

In the preparation of the hammermilled particles, 1-½ inch (3.81 cm)-wide PB strips were cut from 1-½ inch (3.81 cm)-thick recycled PB panels and broken down into particles by a Blue Streak hammermill (Prater Pulverizer Company, Chicago) of 25 HP at 1765 rpm, without screening. It took approximately 45 s to break down 10 PB strips about 30 inches long.

Sample Characterisation

All chipped furnishes were ground using a Thomas Wiley Laboratory Mill (Thomas Scientific, USA) with a 2-mm screen before further processing.

After the steam explosion, the airborne free formaldehyde content of the recycled materials was tested three times according to the National Institute of Occupational Safety and the Health Analytical Method 3500 (NIOSH 1994) at the outlet of the steam explosion facility.

The free phenol content of the recycled OSB was tested by means of gas-liquid chromatography in accordance with the Standard Test Method for Phenols in water according to ASTM D2580-06 (ASTM 2006). In preparation of samples for gas chromatography-mass spectrometry (GCMS) test, steam explosion treated 25 g recycled OSB fibers were mixed with 200 mL of distilled water and stirred for 5 minutes. The flask with the mixture was kept still in a 4 °C refrigerator for 24 hours. Then the solution with free phenol was obtained by discarding deposit OSB wood fibers. For GCMS test, 0.2 g of obtained solution was mixed with 10 mL of CH_3OH and 1 mL of the mixed solution was used for GCMS test with triplicates. A Hewlett-Pakard (HP) Model GC 5890 Series II gas chromatograph equipped with an HP 5971 series mass selective detector and an HP 7673 GC autosampler was used for the analysis. A DB-5 fused silica capillary column (J&W Scientific, Folsom, CA) was used. The column temperature was initially held at 50 °C for 4 min, then the temperature was raised to 220 °C at a rate of 5 °C per minute, from 220 to 300 °C at a rate of 10 °C per minute and held for 10 min. The injection volume was 1 μL and syringe size was 10 μL with two minutes solvent delay time. Helium was used as carrier gas. Injector temperature was maintained at 270 °C. The test was performed in the scan mode.

The pH and buffer capacity of the recycled materials was tested according to the procedure described by Wang *et al.* (2006). Briefly, it includes weighing 25 g of wood composite flour and placing the flour in a 500-mL flask equipped with a condenser; adding 200 mL of boiled/distilled water and reflux the mixture for 20 minutes; once the mixture is cool, filtering it under vacuum with an 11-cm Whatman #4 filter paper; then washing the mixture with distilled water; filling the flask to 500 mL; calibrating the pH meter with pH 7.00 and 4.00 standard buffer solutions. To determine the wood composite flour acid buffer capacity, for example, one needs to titrate a 100-mL sample of extractive solution and bring it to a pH of 3.00 with a standard 0.025 N sulphuric acid (H_2S0_4) solution. The initial pH value from titration is reported as the pH of the wood composite flour. The acid buffer capacity, expressed as total milliequivalents (mEq) of acid needed to lower the pH to 3.00, can be calculated as:

(Volume of Titrant x 0.025 N of H_2S0_4 x Volume of Flask x 100 g of OD Wood)/ (Volume of Titrated Sample x Weight of OD Wood Used)

The particle size of the different materials was determined using an image analysis device. About one gram of oven-dried material was sampled from each type of furnish. Black and white images were taken, and the intensity and/or colour differentiation were used to distinguish the wood from background. All the particles smaller than 0.03 mm^2 were filtered out. The weighted average fiber length of the steam-exploded materials was measured by a Bauer-McNett Classifier according to TAPPI Standard T233 CM-06, and the average fiber length was determined according to the Tasman Method (Tasman 1972).

Statistical Analysis

The means and standard deviations of the particle size were calculated. The data were analyzed statistically using Duncan's multiple range tests through SAS® 9.3 (SAS, Cary, NC).

RESULTS AND DISCUSSION

Steam Explosion of MDF, PB, and OSB Panels

After the steam explosion, the fibers prepared from recycled MDF, PB, and OSB panel chips were measured for length using a Bauer McNett Classifier. The test results of the fiber length distributions are presented in Table 3.

Table 3. Fiber Length Distribution of Steam-Exploded Recycled Materials

Fiber type	Tyler meshes screens number (mm)					Weighted average fiber length (mm) $L=\frac{w_1l_1+w_2l_2+\ldots w_5l_5}{W^*}$
	14 (1.19)	28 (0.595)	48 (0.297)	200 (0.074)	< 200 (0.074)	
	W_1 (%)	W_2 (%)	W_3 (%)	W_4 (%)	W_5 (%)	
MDFF	1.78	31.1	34.2	3.34	29.6	1.127
MDFS	1.68	14.2	29.6	4.0	50.5	0.792
PBS	14.0	16.2	17.4	2.3	50.1	1.045
OSBS	17.5	18.6	16.0	2.4	45.6	1.170
Fiber length (l_i) (mm)	3.0 (l_1)	1.9 (l_2)	1.2 (l_3)	0.4 (l_4)	0.2 (l_5)	

MDFF MDF fibers obtained from a mill
MDFS Fibers prepared from recycled MDF panels by steam explosion
PBS Fibers prepared from recycled PB panels by steam explosion
OSBS Fibers prepared from recycled OSB panels by steam explosion
$*W=W_1+W_2+W_3+W_4+W_5$

The average fiber length of steam-exploded MDF chips was 30% shorter than the average MDF fiber sampled from the same MDF mill. More than 50% of the fibers subjected to steam explosion passed through a 200-mesh screen, compared to 30% of the regular MDF fibers.

The average fiber length of the recycled PB was longer than that of the recycled MDF fiber, but shorter than that of the fresh MDF fiber from the MDF mill. More recycled PB fibers were collected in the 14-mesh, or 3 mm, category than were MDF fibers from the MDF mill. This may show that the steam explosion condition is more suitable for processing PB, which is of use to processing of recycled PB for MDF manufacturing.

The average fiber length of recycled OSB was longer than that of the recycled MDF and PB fibers, and MDF fibers from industry. More recycled OSB fibers were collected in the 14 mesh, or 3 mm, category than were the MDF fibers from the mill. This shows that recycling OSB by steam explosion may have potential to produce better quality fibers, in terms of fiber length, if the right steam explosion condition is used.

Because the fiber length of fibers used in MDF production is relative constant, based on the information above, considering possible variation in wood species used, it can be concluded that steam explosion appeared to reduce the fiber length of recycled steam-exploded MDF. Because a relatively large amount (45.6%) of fine fibers (< 200 mesh) was obtained for all recycled materials, further research should be focused on how to reduce the amount of fine fibers of steam-exploded recycled materials. The impact of large or long fibers (> 14 mesh) of the recycled furnishes should also be investigated.

Chemical Impregnation of MDF and PB Panels

It was found that with pure tap water impregnation and treated with steam at 113 °C for 20 min, MDF samples were softened and swelled but did not separate (Fig. 1a). It was also found that the addition of 0.5% BTCA solution was very effective in breaking down the recycled MDF panels, as the samples were completely disintegrated (Fig. 1b).

Concerning PB, as with MDF, it was found that with pure tap water impregnation and steam treatment at 113 °C for 20 min, the PB samples swelled, and the laminated paper could be separated from the used PB (Fig. 1c, upper sample), but the particles did not separate. The impregnation of PB samples with 0.5% BTCA solution and then steam treatment was very effective at breaking down the recycled PB panels. A complete disintegration (Fig. 1c, bottom sample) of the PB was observed.

A B C

Fig. 1. Chemical-impregnated MDF and PB

Sample Characterisation

Free formaldehyde and VOC emissions

When measured at the material collection cyclone outlet, the free formaldehyde emission of the MDF panels subjected to steam explosion was 1 ppm, according to Drager colorimetric detector tubes (Zefon, USA), slightly higher than the permissible exposure limit for formaldehyde vapors established by the Occupational Safety and Health Administration (OSHA 2014) of the USA, which is 0.75 ppm as an 8-h time-weighted average.

Free phenol content of recycled OSB panel

The free phenol content of the recycled OSB panels was about 0.01%, which was much lower than the < 5% specified by all major resin manufacturers in the USA (Lawson and Petri 2014), indicating that recycling OSB into panels will not raise environmental issues in terms of phenol emissions.

pH and buffer capacity of different materials

Listed in Table 4 are the pH values and buffer capacities of different wood panel materials. From un-resinated fresh particles (FP) to hammermilled recycled PB furnishes (PBSE), the pH increased slightly. From fresh particles (FP) to steam-exploded PB furnishes (SEP) and to BTCA-treated PB furnishes (PTWB), the pH value was noticeably increased. This finding was unexpected. From the perspective of the compositions of these wood composite materials, it was expected that the pH of hammermilled recycled PB furnishes would be lower than that of un-resinated fresh particles, since PB is bonded by UF resin, and UF resin is catalyzed or cured under acidic conditions, and that the pH of steam- and BTCA (an acid)-treated PB furnishes would decrease after treatments.

Table 4. pH and Buffer Capacity of Different Materials

Materials	pH	Buffer Capacity (mEq)
Fresh particles (FP)	5.19	10.03
PB Hammermilled (PBSE)	5.21	4.4
OSB raw material (Aspen)	6.23	
OSB before steam explosion (OBSE)	6.59	
MDF before steam explosion (MSE)	5.33	7.3
MDF treated with BTCA (MTWB)	7.51	
PB treated with BTCA (PTWB)	6.39	
Steam-exploded OSB (SEO)	4.29	
Steam-exploded PB (SEP)	5.7-5.81	112.2
Steam-exploded MDF (SEM)	6.72-7.09	107.1

However, this phenomenon can be explained from a dynamic point of view. It is known that UF resin is hydrolysable at least at 100 °C in water solution (Freeman and Kreibich 1968), and UF resin is catalyzed by, normally, NH_4Cl, creating an acidic environment within a hot pressed panel. Under this acidic and hot condition wood panel furnishes will be hydrolyzed and release acids (Lee *et al.* 1994). Similarly, during steam disintegration of MDF and PB, there are at least two hydrolysis reactions. One is the hydrolysis of wood components and another is the hydrolysis of UF resin. The hydrolysis of wood components will reduce wood pH due to the acids released. The hydrolysis of UF resin is more complicated. It is known that UF resin is mostly composed of urea, which consists of ammonia and carbon dioxide. In water solution, ammonia is converted into ammonium hydroxide, NH_4OH, which is a very strong alkaline substance (Poblete and Roffael 1985). When UF resin in a panel is hydrolyzed, some of it will be broken down into urea and formaldehyde. The urea and the NH_4Cl or $(NH_4)_2SO_4$ catalyst will be mainly broken down into ammonia, which turns into ammonium hydroxide and increases the pH of the disintegrated panel furnishes (Freeman and Kreibich 1968; Roffael *et al.* 2009). Therefore, the final pH of steam-disintegrated recycled panel furnishes depends on an equilibrium between the two hydrolysis processes aforementioned. The results obtained in Table 4 may illustrate that UF resin hydrolysis dominated the processes of SEP and PTWB, and that the final pH value of SEP and PTWB increased. Regarding the pH of hammermilled PB (PBSE), the results may indicate that the UF resin and wood component hydrolyses during the hot press were offset by each other such that the pH of hammermilled PB (PBSE) was about the same as that of fresh particles (FP).

The final pH of a disintegrated UF resin-bonded panel furnish may also depend on other parameters, such as time and temperature involved in the treatment. The impact of temperature may be reflected in the pH difference between SEP and PTWB, where the high temperature accelerated the hydrolysis of wood components and thus led to a lower pH of SEP, since SEP was processed at 227 °C. Indeed, Roffael and Huster (2012) also found a similar situation. They observed that within three hours of the hydrolysis, increasing the temperature from 40 to 103 °C did not affect the pH value much, while within 24 h, the pH value of the hydrolyzed UF resin-bonded panel increased with temperature in the range of 40 to 103 °C. Further increasing the hydrolysis temperature from 103 to 150 °C decreased the pH value. In a similar way, the pH differences between different MDF materials observed in the current study can be explained.

Another unexpected result was that the pH of steam-exploded OSB furnishes (SEO) was lower than that of aspen and OSB furnishes not subjected to steam explosion treatment (OBSE). It was expected that the pH of SEO would be higher than that of aspen, the major wood species used in OSB production in Canada, due to the fact that PF resin is alkaline-catalyzed. Unlike what happened on UF resin in recycled PB furnishes, the PF resin was not easy to break down or to hydrolyze (Freeman and Kreibich 1968). Cured PF resins are insoluble and as a result, the pH value of PF resin might not have much impact on that of SEO. Hence, the pH value of SEO was mainly affected by the hydrolysis of the wood components, which led to a decrease in the pH of SEO.

It was difficult to understand why there was not much difference in pH between fresh raw PB materials (FP) and hammermilled PB furnishes (PBSE), while there was a relatively large difference in pH between aspen and chipped OSB furnishes (OBSE). It was suspected that wood species difference (aspen for OSB, black spruce/jack pine/balsam fir (SPF) for PB), resin difference (PF resin in OSB, UF resin in PB), and the interaction between wood species and resins under heat and wet condition would be attributable to the fact mentioned above. Further research is needed to verify this.

From Table 4 one can see that FP had a higher buffer capacity than PBSE and MSE. This indicates that the UF resin present in recycled hammermilled PB and MDF might reduce the buffer capacity of furnishes. However, what caused the buffer capacity difference between PBSE and MSE remains unknown. It was expected that the refining process in MDF production might have had some impact.

After recycled PB and MDF panels were exposed to steam explosion, and their buffer capacity increased exponentially; for PB or SEP it was 112.2, and for MDF or SEM it was 107.1. This may be attributable to the decomposition of UF resin in steam-exploded panel materials. Nevertheless, further research is needed to determine if it is UF resin decomposition or the hydrolysis of wood components that increases the buffer capacity, and how the pH and buffer capacity affect panel production and properties or a combination of both.

Since chipped or hammermilled PB (PBSE) and MDF (MSE) had about the same pH and lower buffer capacity compared to fresh particles, it was expected that recycling hammermilled PB and MDF into panels would increase the UF resin curing speed. Since steam-exploded UF resin-bonded panel materials had high pH and buffer capacity, it was also expected that conventional UF resin and hot press cycles might not be suitable for bonding steam-exploded materials (Gunnells 2000).

Size of particles

Four different types of particles were measured for size distribution: fresh particles from a PB mill (core and face furnishes), hammermilled recycled panel particles (containing face and core layers), and recycled particles from panels impregnated with BTCA solution (containing face and core layers). Table 5 shows the average particle size for each type of material with commercial core furnishes being the largest. Table 6 shows an overview of the particle size distribution.

Table 5. Average Size of Different Particles

Materials	Particle size (mm^2)
Commercial face furnish	1.05 (1.38) A
BTCA treated	1.32 (3.19) B
Hammermilled	3.23 (3.49) C
Commercial core furnish	6.65 (7.65) D

Values in parentheses are standard deviations.
Values in the same column with different letters are significantly different at $\alpha = 0.05$.

Table 6 shows that hammermilled recycled particles included more particles smaller than 4 mm^2 than did fresh core particles. They included fewer particles smaller in size than 1 mm^2 but more in sizes greater than 2 mm^2 than did fresh face particles. They had fewer particles smaller in size than 1 mm^2 and more in the sizes greater than 2 mm^2 compared to particles from recycled panels impregnated with BTCA solution. This was unanticipated because it was expected that hammermilling would further break down particles of all sizes. One of the factors attributable to this unexpected result may be that during the hammermilling process, not all particles were broken down, particularly for those of small sizes in the face layer, due to UF resin bonding. This may also explain why the hammermilled particles were larger on average than the BTCA-treated particles. The difference in average particle size between these four types of particles was significant at a 0.05 level. Due to the fact that in BTCA-treated particles, there were materials for the PB core layer and that the size of hammermilled particles was greater than that of face materials, one should use both BTCA-treated particles and hammermilled recycled particles in the core layer of a reconstituted PB panel.

Table 6. An Overview of the Particle Size Distributions

Particle size (mm^2)	*Core*	Hammermilled	Face	BTCA
1	9	17	65.1	65.7
2	11.4	30.8	21.2	18.6
3	14	21.4	7.1	7
4	12.8	14.5	3.1	3.1
5	9.7	8	1.5	1.5
6	7.6	5.2	0.8	0.7
7	5.4	3.7	0.5	0.8
8	5	2.9	0.3	0.5
9	3.9	1.8	0.2	0.5
10	2.2	1.4	0.1	0.3
50	18.4	4.1	0.2	1.3
75	0.4	0.1	0	0.1
100	0.1	0	0	0

CONCLUSIONS

1. Steam explosion treatment reduced the fiber length of recycled PB and OSB material, and it might reduce the fiber length of recycled MDF material too. During the steam explosion process, the free formaldehyde emission of recycled MDF furnishes was 1 ppm, slightly higher than the permissible exposure limit for formaldehyde vapors established by the OHSA. The treatment increased the pH and buffer capacity of recycled UF resin-bonded panel furnishes, while it decreased the pH of PF resin-bonded recycled OSB panel furnishes.
2. Impregnation using 0.5% BTCA solution together with a thermal treatment effectively disintegrated the recycled UF resin bonded MDF and PB panels. The treatment increased the pH of recycled UF resin-bonded panel furnishes.
3. Hammermilling recycled UF resin-bonded PB was less likely break particles down into sizes less than 1 mm^2.
4. The free phenol content of a recycled PF resin-bonded OSB panel was about 0.01%, which was much lower than the < 5% specified by all major resin manufacturers in the USA, indicating that recycling PF resin bonded OSB into panels will not raise environmental issues in terms of phenol emissions.

ACKNOWLEDGMENTS

This manuscript is cataloged as FWRC-FP765, Forest & Wildlife Research Center, Mississippi State University, MS 39762-9820. This research was conducted at FPInnovations, Canada. The contributions of the staff of FPInnovations are greatly appreciated. The publication of this work was supported by the MSU-FWRC, USDA, NIFA, and the McIntire-Stennis Cooperative Forestry Research Program. The authors are grateful for the financial support from these institutions.

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Article submitted: July 21, 2014; Peer review completed: September 7, 2014; Revised version received and accepted: October 23, 2014; Published: October 28, 2014.