The Effect of Natural Fibres Mercerization on Natural Fibres/Polypropylene Composites: A Study of Thermal Stability, Morphology and Infrared Spectrum

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ABSTRACT
This research was carried out to evaluate how alkali treatment of selected natural fibres (kunai, luffa, betel nut, banana and rice straw) influences thermal properties, morphological properties and infrared spectrum properties of the composites made from polypropylene reinforced with natural fibres. Composites were made by compression moulding technique using hydraulic hot press. Composites were characterized by thermo-gravimetric analysis (TGA) to establish their thermal stability. The infrared spectrum of both treated and untreated natural fibres/PP composites were studied using Fourier transforms infrared spectrometer (FTIR). Infrared spectroscopy is sensitive to the presence of chemical functional groups in the composites. A functional group is a structural fragment within a molecule. Scanning electron microscopy (SEM) was used to investigate the morphology of composites. Increase in the thermal stability and better fibre-matrix compatibility of the composites were noticed in the treated fibres. Thermal stability of all the reinforced natural fibres was found to be around 205°C. Decomposition of both cellulose and hemicellulose in the fibres took place at 310°C and above, whereas the degradation of reinforced fibre composites took place above 430°C. After the chemical treatment, the FTIR results showed the reduction of OH bonds for all natural fibre composites used in this study. Mercerization had successfully modified the structure of natural fibres and these modifications improved the thermal stability of the composites by promoting better fibre-matrix bonding.

INTRODUCTION

Natural fibre reinforced polymer matrix composites have attracted great attention because of properties such as biodegradability, non-toxicity, low density, non-abrasiveness, eco-friendliness, cost-effectiveness and sustainability (Kalia et al., 2013). The main drawback of natural fibres is their hydrophilic nature, which lowers their compatibility with hydrophobic polymer matrices. The presence of a natural waxy substance on the fibre surface, and hydroxyl groups lead to ineffective fibre-polymer matrix bonding and poor surface wetting. High moisture absorption of the fibre causes swelling and plasticizing effect, resulting in dimensional instability (Mohanty et al., 2001). The interfacial adhesion between natural fibres and polymer matrices has often been a vital issue in several different natural fibres reinforced polymer matrix composite systems. A strong bond in the interface is necessary to achieve high performances of composites. It has been established that the mechanical, thermal and acoustical performances of the composites depends not only on the properties of the principle components but also on the nature and strength of the interface. Therefore, a number of chemical and physical surface modification studies on a natural fibre have been crucial for understanding and enhancing the interfacial adhesion between the natural fibres and the polymer matrix, which further improve the composite performances (Lee et al., 2008). Interface is an infinitesimally thin section between fibre and matrix phases, whereas interphase is an interfacial region with finite volume and distinct physical properties or gradients in properties (Felix, 1993). An interface is formed if the fibre or matrix phases influence a region of the other phase in a manner that alters its chemical or physical structure. The interfacial properties can be improved by giving appropriate modifications to the components, which give rise to changes in physical and chemical interactions at the interface (John and Anandjiwala, 2008). The surface modification typically involves one of four methods,
such as physical, chemical, mechanical and physical-chemical. Physical treatments include plasma, corona, chemical grafting by direct condensation, vacuum ultraviolet, x-radiation, and laser. Chemical treatments include silanes, alkali, acetylation, acrylation, benzylation, maleated coupling agents, isocyanates, and peroxidase. Mechanical methods involve rolling or swaging may damage the fibres. The physical-chemical methods involve solvent extraction of surface gums and other soluble components of the fibres (Satyanarayana et al., 2009). The main characterization techniques usually applied to assess the occurrence and the extent of the modification include Fourier transform infrared (FTIR), elemental analysis, inverse gas chromatography (IGC), contact angle measurements, x-ray photoelectron spectroscopy (XPS) and scanning electron microscopy (SEM). Recently, new emerging techniques, such as cross-polarization, magic-angle-spinning, nuclear, magnetic resonance, atomic force microscopy (AFM), angle take-off photoelectron, secondary ion mass spectrometry and confocal fluorescence microscopy have started to be used in this field (Gandini and Belgacem, 2011). A better understanding of chemical composition and surface adhesive bonding of natural fibre is necessary for developing reinforced natural fibre composites. The reinforcing efficiency of natural fibre is related to the nature of cellulose and its crystalline structure. The main constituents of natural fibres are cellulose (α-cellulose), hemicellulose, lignin, pectin, water soluble substances and waxes. There are various factors which affect the properties of natural fibres such as maturity, harvest, variety, climate, decortication, disintegration, fibre modifications, retting degree and spinning and carding. Natural fibres are amenable to chemical modifications due to the presence of hydroxyl groups. Surface characteristics such as wetting, adhesion, surface tension, or porosity of fibres can be improved with chemical treatments of fibres. This research was focused on alkali treatment on different kinds of natural fibres such as kenaf, luffa, rice straw, banana and betel nut, and its effect on thermal stability, functional group changes in infrared spectroscopy and morphological properties.

**Alkaline Treatment:**

The treatment on natural fibres by sodium hydroxide (NaOH) is widely being used to modify the cellulosic molecular structure. Alkali treatment leads to the increase in the amount of amorphous cellulose at the expense of crystalline cellulose. During alkali treatment the removal of hydrogen bonding takes place in the fibre network structure. Alkali treatment of natural fibres, also called mercerization, is the most common method to produce quality fibres (Ray et al., 2001). According to ASTM D1965, the mercerization is a process that allows vegetable fibres to interact with a low concentrated aqueous solution from a strong base (Bledzki and Gassan, 1999). This process indirectly produces swelling due to changes in the fine structure, morphology, dimension and mechanical properties (Bledzki and Gassan, 1999). During alkali treatment, the alkali sensitive hydroxyl (OH) groups present in the natural fibre molecules are broken down, which then react with water molecules (H-OH) and move out from the fibre structure. The remaining reactive molecules form the fibre cell –O-Na groups between the cellulose molecular chains (John and Anandjiwala, 2008). Due to this, hydrophilic hydroxyl groups are reduced and the fibres’ moisture resistance property is increased. The treatment also takes out a certain portion of hemicelluloses, wax, lignin, pectin, and oil covering materials (Li et al., 2007). Alkali treatment leads to fibrillation, which breaks the composites’ fibre bundle into smaller fibres and reduces the fibre diameter. Therefore, increase in the aspect ratio of the fibre might lead to enhanced interface adhesion between fibre and matrix due to higher surface roughness (Maiti et al., 2014). Mechanical and thermal behaviours of the composites are improved significantly by this treatment. Treated fibres have lower lignin content than untreated fibres (Kabir et al., 2012). Chemical treatment partially removes the wax and oil that cover materials and distension of crystalline cellulose order (Kabir et al., 2012).

**MATERIALS AND METHODS**

Rice straw stem fibres are widely available in different parts of the world and can be used as very low-cost reinforcements for composites. The rice straw stem fibre is mainly consisting of carbohydrate components such as hemicellulose, cellulose and lignin (Cheng et al., 2004). Luffa sponge is commercially viable and environmentally acceptable bio-material derived from fruit of *luffa cylindical* plant and has recycling capability and triggered biodegradability (Shen et al., 2012). Betel nut (*areca catechu*) fruits are covered with shells and the shell of each betel nut fruit produce nearly 2.50 – 2.75g of fibre. Betel nut fibre is biodegradable and an inexpensive raw material (Chakrabarty et al., 2012). Banana fibres (*musa sapientum*) obtained from the pseudostem of the plant (Guimarães et al., 2009). Kenaf (*hibiscus cannabinus, L Malvaceae family*) is an herbaceous annual plant, as it completes its lifecycle within a year. It can be grown under a wide range of weather condition. Kenaf is clearly known as a cellulosic source with economic and ecological advantages. It has low density, high biodegradability, non-abrasiveness during processing and certain other superior mechanical properties (Nishino et al., 2003). All the above mentioned fibres were obtained from local sources in Kota Samarahan, Sarawak, Malaysia.

Rice straw stem fibres, luffa, kenaf, banana and betel nut fibres were washed at room temperature with water-soap solution at the concentration of 8g per litre (Ramzy et al., 2014). Which were then rinsed with tap
water to remove any dust or impurities present in the fibres (Ramzy et al., 2014). The fibres were then soaked in 5% weightage of sodium hydroxide solution at 25°C for 2 days. Finally, fibres were rinsed with distilled water at room temperature and dried in oven (ECOCCELL EC55 with natural air convection, from MMM group) at 60°C for 24 hours. PP is the most versatile and popular thermoplastic polymer used for manufacturing economical materials for applications that do not require superior mechanical properties. PP is a polymer of propylene, meaning that its polymer chain has a methyl (CH$_3$) group attached to half of the carbon atoms along the chain. Polypropylene (PP) is a polyolefin a polymer built from repeating units of simple hydrocarbons. Polypropylene’s basic properties can also be changed a great deal by compounding it with fillers and reinforcements. These modifications and its low density and cost have allowed PP to compete with traditional engineering polymers (Tolinski, 2011). In this research polypropylene pellets supplied by Polypropylene Malaysia Sdn. Bhd. had density of 0.9g/cm$^3$ and melt flow rate of 1.6g/10min at 230°C.

<table>
<thead>
<tr>
<th>Natural fibres</th>
<th>Cellulose (%)</th>
<th>Hemicelluloses (%)</th>
<th>Lignin (%)</th>
<th>Ash (%)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rice straw stem</td>
<td>24 - 46</td>
<td>24 - 28</td>
<td>4 - 6</td>
<td>8 - 16</td>
<td>Yao et al. (2008)</td>
</tr>
<tr>
<td>Luffa</td>
<td>63.0 ± 2.5</td>
<td>11.69 ± 1.2</td>
<td>20.88 ± 1.4</td>
<td>0.4 ± 0.10</td>
<td>Saw et al. (2013)</td>
</tr>
<tr>
<td>Kenaf</td>
<td>72</td>
<td>20.3</td>
<td>9</td>
<td>Faruk et al. (2012)</td>
<td></td>
</tr>
<tr>
<td>Banana</td>
<td>60 – 65</td>
<td>6 – 8</td>
<td>5 – 10</td>
<td>1.2</td>
<td>Satyanarayana et al. (2007)</td>
</tr>
<tr>
<td>Betel nut</td>
<td>α – cellulose 53.20</td>
<td>32.98</td>
<td>7.20</td>
<td>1.05</td>
<td>Hassan et al. (2010)</td>
</tr>
</tbody>
</table>

**Fabrication Of The Composites:**

The study involves 10 (5 chemically treated and 5 untreated) samples that were made with the following manufacturing parameters: a specific gravity of 0.8 and 25/75 by wt% of natural fibres (kenaf, luffa, betel nut, banana and rice straw)/polypropylene. All samples were manufactured by using compression moulding technique. The polypropylene and natural fibres were mixed and loaded into the mould of size 30cm X 30cm X 25 mm. The hot press was preheated at 190°C. pressure was set at 6.89 MPa and the mould was placed between the plates of the hot press. After 30 minutes the mould was cooled by using water as the coolant.

**Characterization Of Composites:**

**Thermal Stability:**

Thermo-gravimetric analysis TGA was conducted on the natural fibres/polypropylene composites to cover the spectrum for both untreated and treated fibre composites. The TGA was performed on TA-60WS workstation analysers (Shimadzu Corp.; Kyoto, Japan) at a heating rate of 10 °C/min. Samples of approximately 30mg were placed in appropriate dish. Specimens were examined under flowing nitrogen (100mL/min) over a temperature range of 30 to 900°C.

**Morphology:**

The morphological studies of the chemically treated fibres were observed using a JEOL JSM-6390LA SEM (Tokyo Japan) with a field emission gun and an accelerating voltage of 5kV to collect images of the surface of composites. The test specimens were sliced and mounted on aluminium stubs with double sided adhesive tape and sputter coated with gold for 5 min to a thickness of approximately 10nm under 0.1torr and 18mA to make the sample conductive.

**Infrared Spectrum:**

Functional groups of composite materials were understood based on the data obtained from FTIR, in the range of 4000cm$^{-1}$ to 400cm$^{-1}$, by using Shimadzu FTIR-8101 spectrometer. The FTIR was used to collect and understand the functional groups of the composite materials. The samples pellets for FTIR spectroscopy were prepared by using approximately 0.5mg of powdered sample that was mixed thoroughly with approximately 100mg of dried powdered potassium bromide (KBr) in a small agate pestle. This mixture was taken in a die of specific dimensions and converted into pellets by applying vacuum pressure. Infrared spectrum was obtained through infrared solution software which presented information in transmittance mode.

**RESULTS AND DISCUSSION**

**Thermal Stability Analysis:**

The thermal characteristics of polypropylene based composites reinforced with natural fibres composites have been presented in Figure 1 (a) to 1 (d). Effects of alkali treatment on the thermal stability of fibres were investigated by means of thermo-gravimetric analysis. Study of thermal degradation of natural fibres is important for the development of natural fibres reinforced polymer matrix composites, in both, service and manufacturing (extrusion, injection moulding, curing and compression moulding). The structural constituents of natural fibres such as cellulose, hemicellulose, and lignin are sensitive with different range of temperatures.
These fibres were exposed to intense heat during composite fabrication. Therefore, a thermal analysis study was conducted to determine the influence of fibres addition into polymer, on thermal stability of composites and to confirm possibility of degradation process during composite fabrication (Arbelaiz et al., 2006).

**Fig. 1:** (a) Thermal analysis curves of untreated kenaf reinforced polypropylene composites.

**Fig. 1:** (b) Thermal analysis curves of treated kenaf reinforced polypropylene composites.

The TGA curve for kenaf fibres shows two degradation steps. The first step had a small drop due to the degradation of the hemicellulose at 305°C whereas the second step had a broad curve at 385°C due the degradation of cellulose and lignin (Brebu and Vasile, 2010). The temperature corresponding to a maximum decomposition rate of kenaf fibres was raised from 466°C to 488°C due to alkali treatment. Alkali treatment reduced non-cellulosic material and increased crystalline structure of the fibres, which enhanced the thermal stability and lead to the temperature shift (Li et al., 2007).

**Fig. 1:** (c) Thermal analysis curves of untreated rice straw reinforced polypropylene composites.

**Fig. 1:** (d) Thermal analysis curves of treated rice straw reinforced polypropylene composites.

The TGA curve of rice straw showed two mass loss steps as also observed by Grozdanov et al., 2006. The similar behaviour of two step thermal degradation occurs for other fibres such as luffa, banana and betel nut. A
marginal improvement in the thermal behaviour of the fibres after NaOH treatment is evident in the Figures 1 (a) to 1 (d). The mass loss that takes place up to 100°C, in all fibres used in this study, may be associated with water loss caused by vapourisation of moisture that was previously present in the fibres (Arbeláiz et al., 2006). The total elimination of water is made difficult by the hydrophilic nature of the fibres, which present structural bound water molecules. A smaller percentage of water loss was observed in chemically treated fibres compared with untreated fibres because the treated fibres have lower hygroscopicity. While the decomposition of lignin was only just initiated, decomposition of other fibre components, such as waxes, glycosides, and pectin took place at this lower temperature range of 110–220°C (Monteiro et al., 2012). Degradation of fibres does not occur until 250°C. Weight loss versus temperature curves in the TGA of untreated fibres had the lowest onset of degradation temperature of 272°C. However, fibres treated with NaOH degraded at higher temperatures, ranging from 310–320°C. All fibres show a considerable mass loss due to decomposition of both cellulose and hemicellulose in the fibres from temperatures 330°C and above. Above 400°C, degradation of fibres can be seen as a result of the break of bonds of the proto-lignin (Faulstich et al., 2006). Lignin decomposes slower than cellulose and hemicellulose components, over a broad temperature range of 200–500°C (Brebu and Vasile, 2010). From the studies conducted, it can be concluded that surface treatment improves thermal performances of natural fibre composites. Table 2 corroborates with the results presented in Figures 1 (a) – (d). Thermal degradation and mode of decomposition under the influence of heat is highly recommended for the optimization of process parameters during fabrication of natural fibres reinforced polymer matrix composites (Pashaei et al., 2011).

### Table 2: Weight loss at different temperatures of pure polypropylene (PP) and PP/fibres composites obtained from TGA with alkali treated (T) and untreated (UT).

<table>
<thead>
<tr>
<th>Samples</th>
<th>100°C UT</th>
<th>100°C T</th>
<th>200°C UT</th>
<th>200°C T</th>
<th>300°C UT</th>
<th>300°C T</th>
<th>400°C UT</th>
<th>400°C T</th>
<th>500°C UT</th>
<th>500°C T</th>
</tr>
</thead>
<tbody>
<tr>
<td>PP + Kenaf</td>
<td>0.8</td>
<td>1.5</td>
<td>1.6</td>
<td>3.8</td>
<td>4</td>
<td>18</td>
<td>20.4</td>
<td>100</td>
<td>100</td>
<td></td>
</tr>
<tr>
<td>PP + Luffia</td>
<td>0.6</td>
<td>1.2</td>
<td>2.8</td>
<td>3.8</td>
<td>4.7</td>
<td>17.3</td>
<td>18.9</td>
<td>100</td>
<td>100</td>
<td></td>
</tr>
<tr>
<td>PP + Betelourt</td>
<td>0.4</td>
<td>1.2</td>
<td>2.5</td>
<td>4.0</td>
<td>5.2</td>
<td>19.8</td>
<td>22.2</td>
<td>100</td>
<td>100</td>
<td></td>
</tr>
<tr>
<td>PP + Banana</td>
<td>0.9</td>
<td>1.4</td>
<td>2.3</td>
<td>4.2</td>
<td>5.2</td>
<td>19.8</td>
<td>22.2</td>
<td>100</td>
<td>100</td>
<td></td>
</tr>
<tr>
<td>PP + Rice straw</td>
<td>0.8</td>
<td>1.2</td>
<td>2.6</td>
<td>4.8</td>
<td>5.8</td>
<td>20.1</td>
<td>21.9</td>
<td>100</td>
<td>100</td>
<td></td>
</tr>
<tr>
<td>Pure PP</td>
<td>0.0</td>
<td>0.0</td>
<td>0.4</td>
<td>6.8</td>
<td>99.8</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### Infrared Spectrum Analysis:

One of the factors that affect the property enhancement of natural fibre reinforced composites is the interactions between the fibre and polymer. In this context, FTIR analysis gives the insight about the interactions between the fibre and polymer. The FTIR spectra of the alkali treated and untreated PP/fibres composites were presented in the mid infrared region 4000 – 400 cm⁻¹. The mid-infrared spectrum (4000 - 400 cm⁻¹) can be approximately divided into four regions and the nature of a group frequency may generally be determined by the region in which it is located. The four regions are, X-H stretching region (4000 - 2500 cm⁻¹), the triple-bond region (2500 - 2000 cm⁻¹), the double bond region (2000 - 1500 cm⁻¹) and the fingerprint region (1500 - 600 cm⁻¹) (Stuart, 2004). The major vibrations in the 4000 - 2500 cm⁻¹ region are generally due to O-H, C-H and N-H stretching. The most useful diagnostic bands to determine the presence of methyl or methylene groups in a sample are the C-H stretching vibrations that these groups exhibit. Triple bond stretching absorptions fall in the 2500 - 2000 cm⁻¹ region because of the high force constants of the bonds. C≡C bond absorb between 2300 and 2050 cm⁻¹, whereas the nitrile group C≡N occurs between 2300 and 2200 cm⁻¹. The principal band in the 2000 - 1500 cm⁻¹ region is due to C≡C and C≡O stretching. One of the more useful group wavenumbers for alkenes is the stretching of the C≡C double bond. The general range of this vibration is 1680 - 1630 cm⁻¹. The spectrum of a molecule may have a hundred or more absorption bands present, but there is no need to assign the vast majority. The spectrum can be regarded as a fingerprint of the molecules in this region 1500 - 650 cm⁻¹, which is referred to as the fingerprint region. The lignocellulosic fibre consists of bundles of hollow cellulose fibrils. The cell walls of fibres are reinforced with spirally orientated cellulose in the hemicellulose and lignin matrix. The constituents at the outer surface of the cell wall are a layer of lignocellulosic material and waxy substances that bond the cell to its adjacent neighbours (Jacob et al., 2005). The FTIR spectrum of the studied polypropylene/natural fibre composites showed various bands at aliphatic hydrocarbon. An aliphatic hydrocarbon is any hydrocarbon that does not contain an aromatic ring. There are three major families of aliphatic hydrocarbons, alkanes, alkenes, and alkynes (Smith, 1999). The simplest type of hydrocarbon is called an alkane. Alkanes consists strictly of carbon-carbon and carbon-hydrogen (C-C and C-H) single bonds. The alkene is any molecule that contains a carbon-carbon double bond (C=C) and it also called as olefins. The alkynes molecule contains a carbon triple bond, C≡C.
Spectra in Figure 2 (a) to 2 (d) show the differences between bands before and after alkali treatment of banana and luffa fibres. For untreated fibres the identification of spectral lines reveal lines representing the hydrogen bonded stretching bands on OH groups in the region 3400 cm\(^{-1}\) and in the region of 1030 – 1150 cm\(^{-1}\). The absorptions can be attributed mainly to the carbohydrates (cellulose and lignin), including C-O-C and C-O stretch (primary and secondary hydroxide groups) and bonds belonging to the glucoside linkage and possibly to lignin since lignin has hydroxyphenyl, guaiacyl and syringyl groups, which are aromatic compounds (Guimarães et al., 2009). The bands near 1650 cm\(^{-1}\) can be assigned to conjugated carbonyl present in typical lignin group. Hemicellulose groups include aliphatic carboxylic acids and ketones. The vibration of aliphatic carboxylic acids and ketones lead to the unconjugated C=O stretching in the bands region of 1725 cm\(^{-1}\). The alkali treatments are used to remove lignin and hemicellulose. The spectral changes often involve variation in the band intensity. The major change in the FTIR spectra of the fibres owing to the alkaline treatment was the reduction of occasional disappearance of typical hemicellulose and lignin bands at 1095.57 cm\(^{-1}\), 1240.23 cm\(^{-1}\), 1371.39 cm\(^{-1}\), and 1456.26 cm\(^{-1}\) that showed same result by Sim et al. 2012. It attributed to CH bending and wagging vibration and CH\(_3\) rocking vibration in PP and C-O and C-C stretching vibration in biomass, CH\(_3\) asymmetric bending vibration in PP and C-H deformation vibration in lignin and carbohydrates (Părpăriţă et al., 2014). The major change in the spectra of the fibres owing to the alkaline treatment was the reduction of the peak centred at 1729-1742 cm\(^{-1}\), attributed to the carbonyl group (C=O) stretching. This disappearance is a consequence of the extraction of hemicellulose and lignin. Other changes include the disappearance of C-H stretching band at 2953.02 cm\(^{-1}\), signifying a reduction in aliphatic fraction of waxes (He et al., 2008). This study confirms that the reduction of the hemicellulose and lignin contents after fibre chemical treatment.

Morphological Analysis:

Scanning electron micrographs of PP-luffa and PP-Betel nut composites at 25wt% were shown in Figure 3 and 4. Figure 3(a) and 4(a) is without alkali treatment and Figure 3(b) and 4(b) is with alkali treatment. In Figure 3(a), fibre de-bonding and fibre fracture are clearly seen in the micrograph.

From Figure 3(a) and 4(a) it can be inferred that the composites with unmodified natural fibres presented few regions with high adhesion and many regions where the fibres were released from the matrix, that is, the adhesion was not homogeneous all over the material. The alkaliized composites showed improved fibre matrix adhesion in Figure 3(b) and 4(b). No visible gap was observed between the fibre and matrix. There were significant differences in the fibre morphology after NaOH treatment. Generally adhesion was better than in the composites reinforced with unmodified natural fibres. A similar result was obtained with henequen fibres by Valadez-Gonzalez et al. (1999).
Conclusions:
Mercerization of natural fibres effectively changes the surface topography of the fibres. The removal of surface impurities on natural fibres may be an advantage for fibre to matrix adhesion. The improvement in fibre/matrix interfacial adhesion was larger when mercerized fibres where used as matrix reinforcement, compared to that of untreated fibres, as confirmed by SEM images. Alkalized natural fibres/polypropylene composites showed the higher thermal stability due to the increased fibre matrix interaction. The mercerization of natural fibres by sodium hydroxide was confirmed by FTIR analysis by the reduction of hydroxyl bands. Further, alkali treatments were shown to have resulted in degradation and dissolution of lignin and hemicellulose to a different extent.

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REFERENCES


