

Vol. 11, No. 4, pp. 165–184, 2018 DOI: 10.14513/actatechjaur.v11.n4.470 Available online at acta.sze.hu

# **Methods of Vector Control for Induction Motors**

# L. Varga<sup>1</sup>, M. Kuczmann<sup>2</sup>

<sup>1</sup>Audi Hungaria Zrt, Department of Petrol Engines Audi Hungária út 1, 9027, Győr, Hungary E-mail: mail@vargalaszlo.com

<sup>2</sup>Széchenyi István University, Department of Automation Egyetem tér 1, 9026, Győr, Hungary E-mail: kuczmann@sze.hu

Abstract: This paper presents the electrical and mathematical models of the three phase asynchronous motors along with the introduction of the field-oriented control model as well as the vector transformations needed for the introduction of the above mentioned terms. The objective of the present paper is to introduce the space vectors and how to build the field-oriented control for a given induction motor drive as well as the transformations and the modell of field oriented control.

Keywords: induction motor, vector control, Field Oriented Control

## 1. Introduction

The asynchronous motor drives are one of the most popular drive types of the industrial environment. Concerning its construction it is a simple drive without too many components, and it is also robust. Beside its positive electrical and mechanical parameters, it can also be operated easily and cost efficient way. The digital revolution in the last 30 years opened several new possibilities in the electronics such as the efficient speed control or the data collecting and analyzing systems. In the Fig. 1 the position of the asynchronous motors can be seen in the world of the electrical motors (the offset non related to the topic are not divided into further parts).

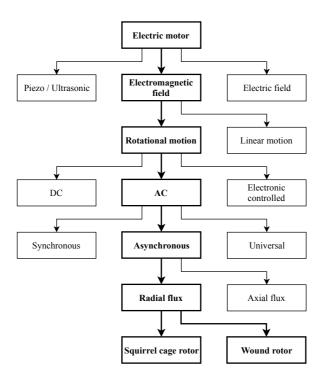


Figure 1. Type of the electrical motors.

Fig. 2 shows the lineage of the field-oriented model from the tree of the variable frequency drive [1].

## 2. Model of the Asynchronous Motor

In the following sections, one after the other, the models of the rotating asynchronous motors are presented.

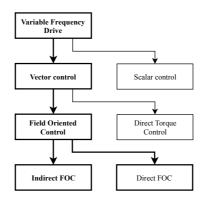


Figure 2. Tree of the Variable Frequency Drive.

### 2.1. Ideal Transformer (ITF) voltage model

The simplest way is when a large air gap transformer is considered - per phase – i.e. the asynchronous motors are ideal for a 1:1 reduced transfer. In this case during the reduction – in contrast with the normal transformer model – the so called efficient turn number of the transfer should be counted with (it is assumed in this case).

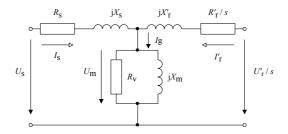


Figure 3. ITF model of asynchronous motor (one phase) [2].

L. Varga and M. Kuczmann - Acta Technica Jaurinensis, Vol. 11, No. 4, pp. 165-184, 2018

The equations of the model are the following (the trivial correlations are concerned known) [3, 8]:

$$s = \frac{n_1 - n}{n},\tag{1}$$

$$U_{\rm m} = 4.44 f_{\rm r} N_{\rm r} \varepsilon_{\rm r} \Phi, \qquad (2)$$

$$U_{\rm s} = I_{\rm s}R_{\rm s} + I_{\rm s}jX_{\rm s} + U_{\rm m},\tag{3}$$

$$U_{\rm mr} = sU_{\rm m} = I'_{\rm r}R'_{\rm r} + I'_{\rm r}jsX'_{\rm r} + U'_{\rm r}, \tag{4}$$

then (4) can be divided by the slip (as it is an ideal transformer), and the result is the stator voltage equation coincides with the voltage equation from the rotating side with the slip so the two substituting sides can be joined together, i.e.

$$U_{\rm m} = I_{\rm r}' \frac{R_{\rm r}'}{s} + I_{\rm r}' j X_{\rm r}' + \frac{U_{\rm r}'}{s}.$$
 (5)

Here s is the slip,  $n_1$  is the speed of the stator field, n is the rotor speed of the rotating machine,  $U_s$  is the primer voltage,  $U_m$  is the inside voltage of the stator side,  $f_r$  is the frequency of the rotor field,  $N_r$  is the turn number of the rotor coil,  $\varepsilon_r$  is the efficiency factor of the rotor side,  $\Phi$  is the flux,  $L_s$  is the leakage inductance of the stator,  $L_r$  is the inductance of the rotor (reduced),  $R_s$  is the coil resistance of the stator,  $R_r$  is the reduced coil resistance of the coil,  $X_s$  is the coil leakage reactance of the stator,  $X_r$  is the reduced coil target reactance of the rotor and the  $U_{mr}$  is the slip-fold inside voltage of the stator side, respectively.

### 2.2. Park-vector model

The model represented in the prior section can give answer for only a few basic questions, so we need to generate the Park-vector form heading from the voltage equation. Look at first the voltage equations of the stator – they can be used for the rotor – with the condition that the coil resistance is concerned to be equal,

$$u_{\rm sa} = U_{\rm sa} \cos\left(\omega t\right),\tag{6}$$

$$u_{\rm sb} = U_{\rm sb} \cos\left(\omega t - 120^\circ\right),\tag{7}$$

$$u_{\rm sc} = U_{\rm sc} \cos\left(\omega t - 240^\circ\right). \tag{8}$$

Equations (6) – (8) are time functions and they do not describe the spatial arrangement but they describe the phase difference between the sine signals. Here  $U_{sa} = U_{sb} = U_{sc}$  are the amplitude of the time functions,  $\omega = 2\pi f$  describe the angular frequency, and t is the time. These time functions describe all phase voltages in stator winding and can be rewritten as the sum of the voltage drop in stator winding and the induced voltage of the flux change which are described by equations (9) – (14) (later the notation of the time t is left, consequently).

To show this expressively Fig. 4 can be used, which shows conceptual circuit diagram of a squirrel cage induction motor. Of course, a similar schematic diagram can be drawn in case of a slip-ring induction motor, in which phase-circuits are equal on stator-side of the squirrel cage induction motor, but other equations would come about because of elements used for different phases of the rotor.

L. Varga and M. Kuczmann - Acta Technica Jaurinensis, Vol. 11, No. 4, pp. 165-184, 2018

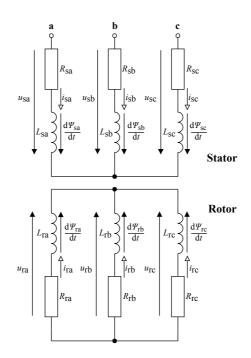


Figure 4. Simple scheme of the Squirrel Cage Induction Motor.

From the Fig. 4, it can be seen the how the stator voltage equations (9) - (11) along with the rotor voltage equations (12) - (14) are generated [3, 6, 9], i.e.

$$u_{\rm sa} = i_{\rm sa}R_{\rm s} + \frac{{\rm d}\,\Psi_{\rm sa}}{{\rm d}t},\tag{9}$$

$$u_{\rm sb} = i_{\rm sb}R_{\rm s} + \frac{\mathrm{d}\,\varPsi_{\rm sb}}{\mathrm{d}t},\tag{10}$$

L. Varga and M. Kuczmann - Acta Technica Jaurinensis, Vol. 11, No. 4, pp. 165-184, 2018

$$u_{\rm sc} = i_{\rm sc} R_{\rm s} + \frac{\mathrm{d}\,\Psi_{\rm sc}}{\mathrm{d}t},\tag{11}$$

$$u_{\rm ra} = i_{\rm ra} R_{\rm r} + \frac{\mathrm{d}\,\Psi_{\rm ra}}{\mathrm{d}t},\tag{12}$$

$$u_{\rm rb} = i_{\rm rb}R_{\rm r} + \frac{\mathrm{d}\Psi_{\rm rb}}{\mathrm{d}t},\tag{13}$$

$$u_{\rm rc} = i_{\rm rc} R_{\rm r} + \frac{\mathrm{d} \Psi_{\rm ca}}{\mathrm{d} t},\tag{14}$$

here  $R_s$  is the stator resistance of any phase coil ( $R_s = R_{sa} = R_{sb} = R_{sc}$ ),  $R_r$  is the rotor resistance of any phase coil ( $R_r = R_{ra} = R_{rb} = R_{rc}$ ),  $i_{sa}$ ,  $i_{sb}$  and  $i_{sc}$  are the currents of the stator coils,  $i_{ra}$ ,  $i_{rb}$  and  $i_{rc}$  are the currents of the rotor coils,  $\Psi_{sa}$ ,  $\Psi_{sb}$  and  $\Psi_{sc}$  are the flux of the stator phases and  $\Psi_{ra}$ ,  $\Psi_{rb}$  and  $\Psi_{rc}$  are the flux of the rotor phases.

Heading from the phase difference of the stator voltage equations (9) - (11) and introducing the following, complex unit vectors in the phase axis directions:

$$\bar{\boldsymbol{a}}^0 = e^{j0^\circ},\tag{15}$$

$$\bar{\boldsymbol{a}}^1 = e^{j120^\circ},\tag{16}$$

$$\bar{a}^2 = e^{j240^\circ},\tag{17}$$

equations (9) - (11) can be brought to a mutual, the so called Park-vector form [9-11]:

$$\bar{\boldsymbol{u}}_{s} = \frac{2}{3} (\bar{\boldsymbol{a}}^{0} u_{sa} + \bar{\boldsymbol{a}}^{1} u_{sb} + \bar{\boldsymbol{a}}^{2} u_{sc}).$$
(18)

As far as (9) - (11) are substituted into (18), the voltage equation (19) of the stator Park vector fixed to the coordinate system of the stator, can be written, i.e.

$$\bar{\boldsymbol{u}}_{\mathbf{s}} = \bar{\boldsymbol{i}}_{\mathbf{s}} R_{\mathbf{s}} + \frac{\mathrm{d}\bar{\boldsymbol{\Psi}}_{\mathbf{s}}}{\mathrm{d}t},\tag{19}$$

here  $\bar{u}_s$  is the common voltage vector of stator,  $\bar{i}_s$  is the common current vector of stator and  $\bar{\Psi}_s$  is the flux linkage of stator.

Using the analogy of (18) for the rotor, the Park vector form of the rotor fixed to the coordinate system of the rotating part can be obtained as

$$\bar{\boldsymbol{u}}_{\mathbf{r}} = \bar{\boldsymbol{i}}_{\mathbf{r}} R_{\mathbf{r}} + \frac{\mathrm{d}\bar{\boldsymbol{\Psi}}_{\mathbf{r}}}{\mathrm{d}t},\tag{20}$$

here  $\bar{u}_r$  is the common voltage vector of rotor,  $\bar{i}_r$  is the common current vector of rotor and  $\bar{\Psi}_s$  is the flux linkage of rotor.

For the graphic representation of the Park vector can be applied the analogy of equations (19) and (20) and generate a common formula [11]. This equation (21) can be used for the stator and for the rotor too [11].

$$\bar{\boldsymbol{u}} = \bar{\boldsymbol{i}}R_{\rm r} + \frac{\mathrm{d}\bar{\boldsymbol{\Psi}}}{\mathrm{d}t},\tag{21}$$

The interpretation of the equation (21) is represented by the Fig. 5. In this interpretation the  $\bar{i}$  current vector can be used better instead of  $\bar{u}$  voltage vector because further calculations are simpler.

As an example in Fig. 5, three phase (a,b,c) with identical frequency and amplitude are used, where the sine signals are shifted by  $120^{\circ}$  and at  $\omega t = 110^{\circ}$ ,

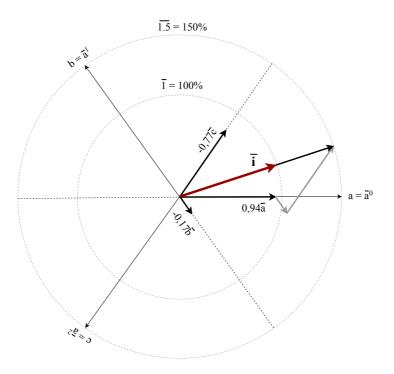


Figure 5. Park-vector scheme.

 $i_a = 0.94$ ,  $i_b = -0.17$  and  $i_c = -0.77$  can be interpreted as the momentary result of the adequated phase power (compared to 1, the whole,  $i_a + i_b + i_c = 0$ ) as well as the  $\bar{i}$  which is the resultive Park-vector current.

The voltage equation (21) is valid for the stator as well as for the rotor, but in both cases, it stands for a coordinate system which is fixed to itself. For the general examination the so called mutual coordinate system is needed. By its help, the voltage and flux equations of the asynchronous machine can be used for the rotor and also for the stator, separately.

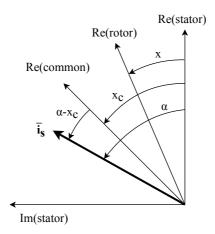


Figure 6. Common coordinates system of the stator.

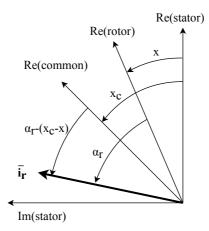


Figure 7. Common coordinates system of the rotor.

According to Fig. 6 [11] the current vector of the stator in the mutual coordinate system is

$$\bar{\boldsymbol{i}}_{s}^{*} = \bar{\boldsymbol{i}}_{s} e^{-jx_{c}},\tag{22}$$

which is transformed by the additional substitution

$$\bar{\boldsymbol{i}}_{\mathbf{s}} = \bar{\boldsymbol{i}}_{\mathbf{s}}^* e^{j\boldsymbol{x}_{\mathbf{c}}},\tag{23}$$

where \* is the sign of the transformation to the common coordinate system.

With this analogy, according to Fig. 7 [11] the current vectors in the mutual coordinate system of the rotor are the following:

$$\bar{\boldsymbol{i}}_{\mathbf{r}}^* = \bar{\boldsymbol{i}}_{\mathbf{r}} e^{-\mathbf{j}(x_{\mathbf{c}}-x)},\tag{24}$$

$$\bar{\boldsymbol{i}}_{\mathbf{r}} = \bar{\boldsymbol{i}}_{\mathbf{r}}^* e^{j(x_c - x)}.$$
(25)

Being back replaced into the equation of (21), and after some mathematical conversions, the asynchronous machine's basic equations with Park-vector can be brought to the following form in the mutual coordinate system (without an asterisk):

$$\bar{\boldsymbol{u}}_{\mathbf{s}} = \bar{\boldsymbol{i}}_{\mathbf{s}} R_{\mathbf{s}} + \frac{\mathrm{d}\boldsymbol{\Psi}_{\mathbf{s}}}{\mathrm{d}t} + \mathrm{j}\omega_{\mathrm{c}}\boldsymbol{\bar{\Psi}}_{\mathbf{s}},\tag{26}$$

$$\bar{\boldsymbol{u}}_{\mathbf{r}} = \bar{\boldsymbol{i}}_{\mathbf{r}}R_{\mathbf{r}} + \frac{\mathrm{d}\boldsymbol{\Psi}_{\mathbf{r}}}{\mathrm{d}t} + \mathbf{j}(\omega_{\mathrm{c}} - \omega)\bar{\boldsymbol{\Psi}}_{\mathrm{r}},\tag{27}$$

where  $\omega$  is the electric angular velocity of the rotor and  $\omega_c$  is the electrical angular velocity of the mutual coordinate-system.

## 2.3. Flux equivalent circuit

The next step of the system analysis is – looking ahead the future phases of the research – the exploration of the flux relationships in the machine model. The transformed power vectors into the mutual coordinate system, also foreshowed in section 2.2. are being used in the flux equations which have their explanation in Fig. 8.

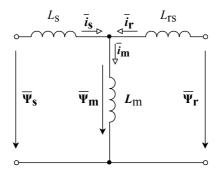


Figure 8. The flux equivalent circuit.

Here  $\bar{\Psi}_s, \bar{\Psi}_m, \bar{\Psi}_r$  are the flux vectors, and  $\bar{i}_s, \bar{i}_m, \bar{i}_r$  are the current vectors. All of them are transformed vectors to the mutual coordinate system of the stator, rotor and the mutual magnetization flux and the current (without \*).

By the help of Fig. 8 [3], it can be seen that the total (F index: "full") inductivity of the stator is

$$L_{\rm sF} = L_{\rm s} + L_{\rm m},\tag{28}$$

and the total inductivity of the rotor is

$$L_{\rm rF} = L_{\rm rs} + L_{\rm m},\tag{29}$$

where  $L_{\rm rs}$  is the leakage inductivity of the rotor,  $L_{\rm m}$  is the inductivity of the mutual magnetization,  $L_{\rm s}$  is the leakage inductivity of the stator.

Using the equations (23), (25) and (28), (29), see in [3], the flux equations belonging to the mutual coordinate system can be generated, which are needed for the extension of the Park-vector model:

$$\bar{\Psi}_{\mathbf{s}} = L_{\mathbf{s}\mathbf{F}}\bar{\boldsymbol{i}}_{\mathbf{s}} + L_{\mathbf{m}}\bar{\boldsymbol{i}}_{\mathbf{r}},\tag{30}$$

$$\bar{\Psi}_{\mathbf{r}} = L_{\mathrm{m}} \bar{\mathbf{i}}_{\mathbf{s}} + L_{\mathrm{rF}} \bar{\mathbf{i}}_{\mathbf{r}}.$$
(31)

The electrical and mathematical models – described until this section – can be used after some condition for the rotating part of the squirrel cage motor. By its help and using the mechanical models of the asynchronous machine – the detailed explanation is not part of this paper, see in [6] - as well as the help of some further transformations, modern and vector field-oriented regulations can be generated.

## 3. Vector control for Induction Motor

The basic idea of the vector control, or its other name, field-oriented control (FOC) is that it the stator's three-phase input is splitted up into 2 orthogonal components that 2 components can appoint one vector. These 2 vectors are the torque and the magnetic flux.

This basic idea goes back to the conception of the former DC motors where the machine's construction lets control the torque (and/or the speed control) as well as the magnetic field independently from each other. This induces the wide dynamic range of the DC machine and also its stability.

On the other hand, the AC machine is non-linear and multi variable system in which the variable parts depend strongly on the outer parameters and on each other parameters as well (such as temperature, magnetic hysteresis, current frequency etc.). Its mathematical model is complicated by retroaction and by cross-effects. It follows from these that the power components which generate the torques cannot be separated with trivial methods.

The regulation of the induction machine with 2 independent components can be attached into a simple loop circle, which can be seen in Fig. 9. From the connections in Fig. 9 and the section 2 the basic scheme of the field-oriented regulations can be composed.

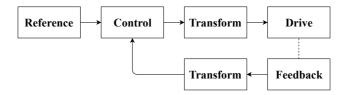


Figure 9. Diagram of the Induction Motor control.

### 3.1. Transformation

The purpose is the application of such a transformation model which makes possible the regulation of time variant parameters as time invariant variables which modifies the Fig. 9 as follows [1, 7].

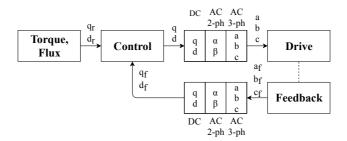


Figure 10. Transformations in the FOC model.

The reference parameters on Fig. 10 are the input Torque  $(q_r)$  and Flux  $(d_r)$  through a control function the Torque  $(q_f)$  and Flux  $(d_f)$  which are coming back from the feedback line, from these are the input parameters of the transformation model [4, 6, 9]. It can be seen from mathematical and electric point of view that the well-manageable and in time non-changing values are the regulatory parameters in the regulation move.

### 3.1.1. Clarke and inverse Clarke transformation

As it is experienced in the section 2 the sine signals can get onto the three phase asynchronous motor coils. These sine signals are in frequency and amplitude consistent but they are shifted in phase to each other with  $120^{\circ}$ .

With the Clarke transformation it is possible to transform vectors  $\bar{a}, \bar{b}, \bar{c}$  (three-phase) vectors to orthogonal  $\bar{\alpha}$  and  $\bar{\beta}$  (two-phase) vectors. These vectors appoint an  $\bar{i}$  vector in the two dimensional space – this is the Park-vector, itself.

The transformation can be conducted with the following steps in form of a vector from the Fig. 5 (furthermore the Park-vector) :

- the axis of phase  $\alpha$  of Park-vector is being turned with the vectors  $\bar{a}$  and  $\bar{i}$  into a x axis of an orthogonal 2 dimensional coordinate system;
- the transformation vector  $\bar{a}$  is itself the vector  $\bar{a}$  of the turned phase axis  $\alpha$ ;
- the transformation vector  $\overline{\beta}$  is cut by the vectors  $\overline{a}$  and  $\overline{i}$  from the y axis of the coordinate system.

In practice the vector  $\bar{\alpha}$  and the Park vector  $\bar{i}$  appoint the Clarke-transform  $\bar{\beta}$  vector, which is presented in the Fig. 11.

The transformation in matrix form is as follow [5, 12]:

$$\begin{bmatrix} \bar{\boldsymbol{i}}_{\boldsymbol{\alpha}} \\ \bar{\boldsymbol{i}}_{\boldsymbol{\beta}} \end{bmatrix} = \frac{2}{3} \begin{bmatrix} 1 & -\frac{1}{2} & -\frac{1}{2} \\ 0 & \frac{\sqrt{3}}{2} & -\frac{\sqrt{3}}{2} \end{bmatrix} \begin{bmatrix} \boldsymbol{i}_{\mathbf{a}} \\ \bar{\boldsymbol{i}}_{\mathbf{b}} \\ \bar{\boldsymbol{i}}_{\mathbf{c}} \end{bmatrix},$$
(32)

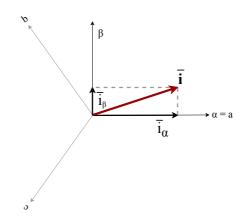


Figure 11. The Clarke transformation.

which is in the feedback line of the Fig. 10 [12], so an inverse transformation is needed in the control line that is the inverse Clarke transformation.

In case of a two phases system conversion to a three phase system, the following matrix is needed [11,12]:

$$\begin{bmatrix} \bar{\boldsymbol{i}}_{a} \\ \bar{\boldsymbol{i}}_{b} \\ \bar{\boldsymbol{i}}_{c} \end{bmatrix} = \begin{bmatrix} 1 & 0 \\ -\frac{1}{2} & \frac{\sqrt{3}}{2} \\ -\frac{1}{2} & -\frac{\sqrt{3}}{2} \end{bmatrix} \begin{bmatrix} \bar{\boldsymbol{i}}_{\alpha} \\ \bar{\boldsymbol{i}}_{\beta} \end{bmatrix}.$$
 (33)

## **3.1.2.** Park and $Park^{-1}$ transformation

After the Clarke transformation the two phase component can be still hardly regulated and it is still alternating. Due to this, after the phase transformation the coordinatesystem's transformation should also be performed which will rotate synchronous with the stator's three-phase rotating field. From these we can receive the needed q Torque and the d Flux in time variant components. These can be easily managed from electrical point of view.

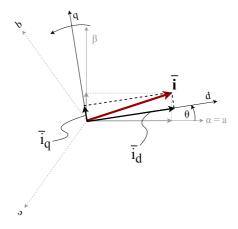


Figure 12. Park transformation [12].

After the presented Park transformation, a synchronous rotating coordinate system will be generated where  $\theta$  is the electrical angle [12]:

$$\begin{bmatrix} \hat{\boldsymbol{i}}_{\boldsymbol{d}} \\ \hat{\boldsymbol{i}}_{\boldsymbol{q}} \end{bmatrix} = \frac{2}{3} \begin{bmatrix} \cos\left(\theta\right) & \sin\left(\theta\right) \\ -\sin\left(\theta\right) & \cos\left(\theta\right) \end{bmatrix} \begin{bmatrix} \hat{\boldsymbol{i}}_{\boldsymbol{\alpha}} \\ \hat{\boldsymbol{i}}_{\boldsymbol{\beta}} \end{bmatrix}.$$
 (34)

In the inverse form of the transformation to the stator coordinate system is returned where again the two phase components appear that are time variant [12]:

$$\begin{bmatrix} \hat{\boldsymbol{i}}_{\boldsymbol{\alpha}} \\ \hat{\boldsymbol{i}}_{\boldsymbol{\beta}} \end{bmatrix} = \frac{2}{3} \begin{bmatrix} \cos\left(\theta\right) & -\sin\left(\theta\right) \\ -\sin\left(\theta\right) & \cos\left(\theta\right) \end{bmatrix} \begin{bmatrix} \hat{\boldsymbol{i}}_{\mathbf{d}} \\ \hat{\boldsymbol{i}}_{\mathbf{q}} \end{bmatrix}.$$
 (35)

### 3.2. Field-Oriented Control

In Fig. 10 the basic model is presented. It can be brought to the following form by the help of the Clarke and the Park transformations:

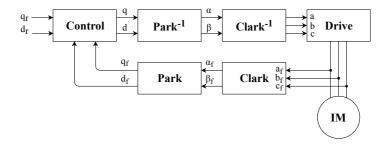


Figure 13. Simple FOC model [3, 5, 9-12].

Fig. 13 represents a simple field-oriented regulation model where the transformations are being built according to the section 3.1., and they can be brought to a mutual matrix shape according to the equation (36) [12]

$$\begin{bmatrix} \hat{\boldsymbol{i}}_{\mathbf{d}} \\ \bar{\boldsymbol{i}}_{\mathbf{q}} \end{bmatrix} = \frac{2}{3} \begin{bmatrix} \cos\left(\theta\right) & \cos\left(\theta - \lambda\right) & \cos\left(\theta + \lambda\right) \\ -\sin\left(\theta\right) & -\sin\left(\theta - \lambda\right) & -\sin\left(\theta + \lambda\right) \end{bmatrix} \begin{vmatrix} \hat{\boldsymbol{i}}_{\mathbf{a}} \\ \bar{\boldsymbol{i}}_{\mathbf{b}} \\ \bar{\boldsymbol{i}}_{\mathbf{c}} \end{vmatrix},$$
(36)

as well as the matrix shape of the inverse transformation is seen as [12]

$$\begin{bmatrix} \hat{\boldsymbol{i}}_{a} \\ \bar{\boldsymbol{i}}_{b} \\ \bar{\boldsymbol{i}}_{c} \end{bmatrix} = \frac{2}{3} \begin{bmatrix} \cos\left(\theta\right) & -\sin\left(\theta\right) \\ \cos\left(\theta - \lambda\right) & -\sin\left(\theta - \lambda\right) \\ \cos\left(\theta + \lambda\right) & -\sin\left(\theta + \lambda\right) \end{bmatrix} \begin{bmatrix} \bar{\boldsymbol{i}}_{d} \\ \bar{\boldsymbol{i}}_{q} \end{bmatrix},$$
(37)

in which  $\lambda = \frac{2\pi}{3}$ .

The presented basic model has several enlargement possibilities for the regulation and also for the feedback line, but these refer to specific models.

# 4. Conclusion

The paper concludes that the vector control for induction motor can be introduced using the scheme of the induction motor and can be prepared transformation matrices for quick calculations of the current vectors. The modells and the calculations will be explained and simulated in the following papers.

# References

- B. Srinu Naik, Comparison of Direct and Indirect Vector Control of Induction Motor, International Journal of New Technologies in Science and Engineering, 2 (1) (2014) pp. 110-131.
- [2] Y. S. Chistyakov, E. V. Kholodova, A. S. Minin, H.-G. Zimmermann, Alois Knoll: Modeling of Electric Power Transformer Using Complex - Valued Neural Networks, Energy Procedia, 12 (2011) pp. 638 - 647. doi:10.1016/j.egypro.2011.10.087
- [3] R. Krishnan, Electric Motor Drives, Modeling, Analysis, and Control, First Indian Reprint, Pearson Education, 2003.
- [4] A. Chikhi, M. Djarallah, K. Chikhi, A Comparative Study of Field-Oriented Control and Direct-Torque Control of Induction Motors Using An Adaptive Flux Observer, Serbian Journal of Electrical Engineering, 7 (1) (2010) pp. 41-55. doi: 10.2298/SJEE1001041C
- [5] Venu Gopal B T, Comparison Between Direct and Indirect Field Oriented Control of Induction Motor, International Journal of Engineering Trends and Technology (IJETT), 43 (6) (2017) pp. 364-369. doi:10.14445/22315381/IJETT-V43P260
- [6] I. Boldea, S. A. Nasar: Electric Drives, 3rd Edition, CRC Press, New York, 2016.

- [7] Kang-Zhi Lui, Masashi Yokoo, Keiichiro Kondo, Tadanao Zamma: New Adaptive Vector Control Methods for Induction Motors with Simpler Structure and Better Performance, Control Theory Tech, Chiba (Japan), 13 (2) (2015) pp. 173–183. doi: 10.1007/s11768-015-4153-z
- [8] K. P. Kovács, Transient Processes of Electrical Machines, Technical Press, Budapest, 1970. (in Hungarian)
- [9] J. Pyrhonen, V. Hrabovcova, R. S. Semken: Electrical Machine Drives Control: An Introduction, 1st Edition, John Wiley & Sons Ltd., Noida, India, 2016.
- [10] B. Akin, Simple Derivative-Free Nonlinear State Observer for Sensorless AC Drives, IEEE/ASME Transactions on Mechatronics, 11 (5) (2006) pp. 634-643. doi: 10.1109/TMECH.2006.882996
- [11] R. De Doncker, D.W.J. Pulle, A. Veltman: Advanced Electrical Drives (Analysis, Modeling, Control), Springer, Germany-Australia-Netherlands, 2011.
- [12] P. Vas: Sensorless Vector and Direct Torque Control, Oxford University Press, New York - Tokyo, 1998.



# The Effect of Filler Orientation and Distribution on the Electrical and Mechanical Properties of Graphene Based Polymer Nanocomposites

# P. K. Rajamani<sup>1</sup>, R. Boros<sup>1</sup>

## <sup>1</sup>Budapest University of Technology and Economics, Department of Polymer Engineering, Műegyetem rkp. 3., 1111 Budapest, Hungary e-mail: rajamani@pt.bme.hu

Abstract: The aim of my proposed study is to develop an alternative production technique of graphene via in-situ exfoliation of graphite in the hosting polymer matrix. The production process is carried out by an alternative production technique for graphene-based top-down polymer nanocomposites called 'pressing and folding' (P&F), via in situ exfoliation of expanded graphite (EG) inside the hosting linear lowdensity polyethylene (LLDPE) matrix. In this way, the properties of the samples containing different wt % of EG is studied as a function of P&F cycles, corresponding to EG exfoliation and distribution throughout the matrix volume. The results confirm that the EG particles were exfoliated completely and increasingly distributed in LLDPE with the number of cycles, and mainly oriented on the plane of the samples. This find was confirmed by a low in-plane resistivity was found for samples prepared between 50 and 150 cycles.

Keywords: In-situ exfoliation, Graphene, Polyethylene, Polymer Nanocomposites, Mechanical Properties

# 1. Introduction

Graphene is a single atom thick sheet of sp2 -hybridized carbon atoms [1]. There has been a rapid rise of interest in the study of the structure and properties of graphene following the first report in 2004 of the preparation and isolation of single graphene layers in Manchester [2]. A single-layer graphene had previously considered as an academic material which would not be possible to remove from the graphite since such 2D crystals would be unstable thermodynamically and/or might roll up into scrolls if prepared as single atomic layers [3]. Research upon the material has now broadened considerably as it was soon realized that graphene might have

other interesting and exciting physical properties such as high levels of stiffness and strength, and thermal conductivity, combined with an impermeability to gases. One obvious application of graphene is in the field of nanocomposites [4] [5] [6] and it has been noticed that the modification of graphene with polymers and the fabrication of graphene-based nanocomposites attracted more and more interests recently.

Polymer based graphene nanocomposites show superior mechanical, thermal, electrical and gas barrier properties when compared to the neat polymer [7] [8] [9] [10]. The most important aspect of these nanocomposites is that all these improvements are obtained at very low filler loadings in the polymer matrix [11] [12]. However, the improvement of these properties depends on the distribution and dispersion of graphene layers in the polymer matrix and as well as interfacial bonding between them. For the efficient utilization of graphite as filler in a polymer composite, its layers must be separated and dispersed throughout the polymeric matrix. Graphite nanoplatelet (GNP), the basic unit obtained by exfoliation of the natural graphite and having a platelet thickness varying from less than 0.34 to 100 nm, is a promising low cost and lightweight alternative to other carbon-based electrically conductive reinforcements for polymer composites [13]. Mainly, two different types of GNPs, such as expanded graphite (EG) and graphite oxide (GO) have been widely used to produce conducting nanocomposites along with improved mechanical properties [14]. There are many studies on these GNP composites based on a range of polymers, including epoxy [15], PMMA [16], polypropylene [17], polyethylene [18] [19], polystyrene [20], nylon [21] and silicone rubber [22].

However, several studies indicated that the dispersion of graphene particles in polymer matrices become challenging due to the interfacial incompatibility [23] of the constituents and for this reason, selection of an appropriate manufacturing technique is essential. There have been various studies discussing the methods of fabricating graphene-based polymer nanocomposites and their corresponding influence on materials properties. For example, Istrate et al [24] prepared a polymer nanocomposite with polyethylene terephthalate (PET) as a matrix and it was reinforced with EG layers solely via melt blending. They have found that with a very little nanofiller loading of 0.07 wt%, the material showed an increase in the elastic modulus higher than 10% and an enhancement in the tensile strength of more than 40% when compared to pure PET.

Another important fabrication method was investigated by Tang et al [25], where they fabricated GO/epoxy composites via solution blending. They prepared different dispersions of GO sheets with and without ball mill mixing and proved that a better GO dispersion in epoxy matrix was obtained after using the ball mill mixing process. Other commonly used fabrication technique includes in-situ polymerization [26] [27], Latex mixing [28] and electropolymerization [29]. Among

the discussed fabrication methods, the solution blending is much better for producing quite homogeneous graphene-based polymer composites because the polymer and the graphene are dissolved in the same or miscible solvent. Also, in-situ polymerization and latex mixing can be a good alternative method for generating homogeneous composites but agglomeration of graphene layers inside the polymer matrix can be a common problem. So, to understand and obtain better graphene dispersion and less agglomeration, there is a need to develop an alternative and simple fabrication technique.

In this present work, a significant effort has been directed to the alternative fabrication of LLDPE/EG nanocomposites and investigate their mechanical and electrical properties. For that purpose, LLDPE are mixed with expanded graphite in a hot press and subjected to 'pressing and folding' (P&F). This work had started with the testing of 10, 20, 50, 100, 150, 200 and 500 cycles of 0.5 wt% of EG content. Later, the experiments are conducted with much higher EG content of 5, 10, 16 and 56 wt%. The main goal is to assess the influence of number of cycles and the filler content on the tensile properties of the prepared polymer nanocomposites. The second part of the manuscript involves in finding the percolation threshold, where the polymer nanocomposites starts to conduct electricity. For this study, four samples with each cycle are made from hot press are tested with the help of twopoint probe method: two for in-plane conductivity and two for out-of-plane conductivity. The third part was to examine the micro-structures of the prepared samples. The samples are analysed in SEM separately with two different broken samples: in tensile machine and cryogenically broken. The final section offers future prospects and conclusions.

# 2. Materials and Methods

## 2.1. Materials Used

The polymer matrix used in this process is linear low-density polyethylene called Flexirene ® MS20A (LLDPE). LLDPE is a commodity polymer, cheap, versatile, with widely uses in a variety of forms. The Graphene Intercalation compound (GICs), commonly called Expanded Graphite is used as a reinforcement of the LLDPE. The Timerex® C-THERM 002 is the particular specification of EG which is being used. EG will improve the properties of the polymer matrix, which is directly related to the degree of dispersion of the nanofillers in the polymer matrix.

### 2.2. Fabrication of polymer nanocomposites

This work takes advantage of an alternative top-down production technique for graphene-based polymer nanocomposites, called "pressing and folding" (P&F), via in situ exfoliation of graphite inside the hosting polymer matrix. In particular, linear low-density polyethylene Flexirene® MS20A (LLDPE) and expanded graphite Timerex® C-THERM<sup>™</sup> 002 (EG) were joined at first, and then gradually mixed by pressing & folding the samples inside a hot-press many times as shown in Fig. 1.

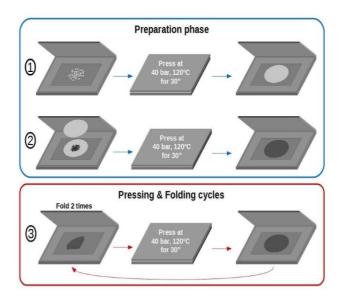


Figure 1. Preparation of samples by pressing and folding

Initially, 1.4 g (0.72g for each discs) of LLDPE discs are prepared using the hot press with the elevated temperature of 120 °C with the pressure of 40 bar and for the duration of 30 seconds. Before incorporating EG into the LLDPE, the pristine LLDPE sample is saved for the benchmark. At first, the minimum filler content of 0.5 wt% to be added between the two PE discs and hot pressed again. After the first cycle the PE plated are folded two times as shown in Fig. 1 and again the sample is hot pressed. Throughout the process, the temperature, Pressure and the press time duration should be kept constant for uniform dispersion of EG. This pressing and folding cycle is continued for different desired cycles. This work had started with the testing of 10, 20, 50, 100, 150, 200, 500 cycles of 0.5 wt% of EG content. Later, the experiments are conducted with higher EG content of 5 wt%, 10 wt%, 16 wt%

#### P. K. Rajamani and R. Boros - Acta Technica Jaurinensis, Vol. 11, No. 4, pp. 185-205, 2018

and 56 wt% (Fig. 2). The hot press configuration pressure of 40bar is applied at a same value for every cycle so that the final sample will have an equal thickness. After the pressing and folding cycles are finished for all the filler content, five dumbbell specimens are made corresponding to ASTM D 638 - 02a.

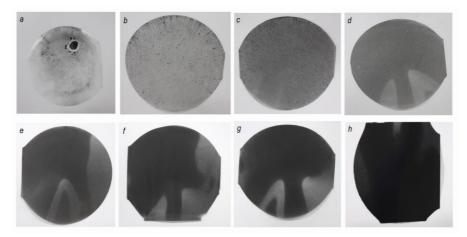


Figure 2. Prepared LLDPE / 0.5 wt% EG samples after a) 1 cycle b) 10 cycles c) 20 cycles d) 50 cycles e)100 cycles f) 150 cycles g) 200 cycles h) 500 cycles

### 2.3. Characterization Techniques

The 'dumbbell' shaped specimen is loaded in the 1 kN load cell of the Instron. As the specimen has a very low thickness ( $\sim$ 0.230mm), the tensile grips should be used to ensure that the specimen is tightly held to the load cell. The rate of speed for the specimen is maintained always at 1 mm/min. With this test, the following parameters can be obtained, such as: tensile strength, elongation at yield, elongation at break, Modulus of elasticity, nominal strain.

The electrical conductivity of the specimen can be obtained with the help of the two-point probe method. From the sample made from the hot press, a strip of approximately 12 X 8 mm of the specimen is trimmed to carry out the electrical testing. For each cycle, four strips are trimmed: two for the in - plane conductivity and two for the out–of–plane conductivity. The strips are individually placed between an aluminium coil prepared, which acts as the extremity. The silver paste can be used between the specimen and the aluminium coil, to keep the aluminium in place and to have an unaffected electric flow between the extremities.

The micro-structures of the prepared nanocomposites were studied analysing the cross-sections of cryogenically broken samples by SEM. The samples were analysed separately with two different broken samples: tensile and cryogenically broken. First, the samples broken using the Instron are analysed through the SEM. The other type of sample can be broken cryogenically with the help of liquid nitrogen. The samples should be sputter coated with conducting material like gold or palladium alloy before introducing in the SEM. This coating is needed to prevent charging of a specimen with an electron beam introduction in conventional SEM.

## 3. Results and Discussion

This section analyses the effect of EG filler content on the LLDPE samples and number of pressing and folding cycles. The section was divided into three parts. The first part and second part are devoted to the mechanical and electrical characterization of the prepared samples, respectively. The last part investigates the dispersion and distribution of EG and exfoliation of graphene in polymer matrix with the help of SEM.

### 3.1. Mechanical Characterization

For the minimum filler content of 0.5 wt%, there are no significant changes were noted to the elastic modulus of pure LLDPE up to 200 cycles, thus the value at 1 cycle ( $135\pm5$  MPa) was taken as reference. The reinforcement due to the EG as a function of cycles can be evaluated by dividing the elastic modulus of each composite by the reference modulus of LLDPE as shown in Fig.3. It was found that the reinforcement (Ec/Em) does not show any proper pattern with the number of increasing cycles.

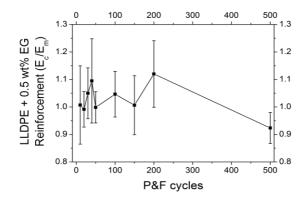


Figure 3. Reinforcement found for sample of LLDPE/0.5 wt% EG as a function of P&F cycle

This non-uniformity in the reinforcement is due to the very small amount of filler that has been added to the LLDPE polymer. So that maintaining an exact 0.5 wt% is a big challenge. The sudden drop in the reinforcement for the 500 cycle is the actual filler content found to be only 0.3 wt% rather than 0.5 wt%. It was expected that the 500-cycle composite will have the highest reinforcement but because of the long process of 500 cycles in the hot press and also because of the small EG content filled in the LLDPE, it does not show a proper pattern (Fig 4). But it's not the case for the LLDPE/10.7 wt% EG and LLDPE/16 wt% EG.

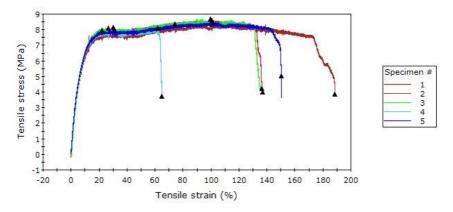


Figure 4. Tensile stress Vs strain for the LLDPE / 0.5 wt % EG at 500 P&F cycles.

Although all nanocomposites exhibit higher modulus than pure LLDPE, they show final failure at much lower stress and strain than the pure LLDPE. In general, the improvement of elastic modulus is attributed to the good dispersion of nanosized particles and good interfacial adhesion between the EG filler and the LLDPE matrix. So that the mobility of polymer chains is restricted under loading. The lower tensile strength of nanocomposites could be due to number of reasons such as weak interfacial bonding between the EG and matrix interfaces, aggregates of graphite nanosheets and nano to micro size process related defects. Fig. 5 shows the stress at break and strain at break as a function of P&F cycles and also the yield stress and strain as a function of P&F cycles. Therefore, the mechanical property of LLDPE / 0.5 wt% EG does not show a good improvement in the P & F technique. It also showed that solution intercalation method shows better results than the melt mixing. As the matrix used here is a thermoplastic polymer (LLDPE), therefore melt mixing along with the new technique called P&F technique was carried out.

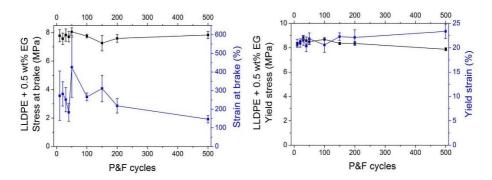
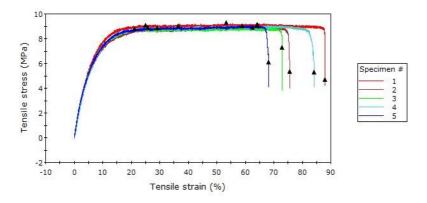


Figure 5. As a function of P&F cycles for the LLDPE / 0.5 wt% EG Nanocomposites a) Stress and strain at break b) Yield stress and Yield strain

The young's modulus is improved than the 0.5 wt% for the 200 cycles. As there were no change in the lower P & F cycles (10,20,100,150), only 200 cycles technique is performed for the 5 wt%. The tensile stress vs the tensile strain is shown in the Fig. 6.





The young's modulus for the LLDPE / 10.7 wt% EG filled nanocomposites showed a good result when comparing with the young's modulus of the pristine LLDPE. But the lower filler concentration (up to 50 cycles) showed approximately equivalent young's modulus to the pristine LLDPE (Fig. 7). It started to gradually increase in the following cycles and reached the maximum of 320 MPa at the 500 cycles.

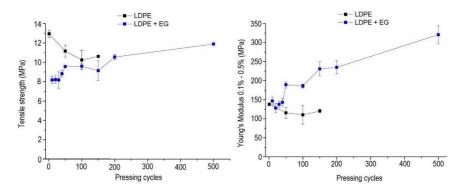


Figure 7. Young's Modulus and tensile strength of the LLDPE / 10.7 wt% EG as a function of P&F cycle

Clearly the presence of EG fillers reduces the ductility of the pristine LLDPE, which shows in the necking without break in the tensile test. Tensile failure depends critically on any localized deformation or agglomeration of EG fillers on any particular area of the specimen. The Young's modulus is measured from the initial region of tensile deformation and indicative of the composite value of the constituent stiffness [30]. Comparing the EG filled composites of higher cycles show a higher modulus than that of the unreinforced LLDPE. Fig. 8 shows the variation of reinforcement (Ec/Em) with EG content for nanocomposites produced by P&F technique.

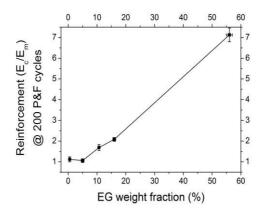


Figure 8. Variation of Reinforcement with EG content

There is a decrease in the reinforcement (Ec/Em) from 0.5 wt% to 5 wt% and that is because of the poor dispersion of the filler content or the presence of defect in tensile tested area. The higher reinforcement is due to the good dispersion of the fillers but it has a more threshold value by adding 56 wt% of EG. The strong interaction between the EG and LLDPE polymer matrix could result from, the wrinkled surface of extremely thin graphite sheets that is capable of mechanically interlocking with polymer chains.

### 3.2. Electrical Characterization

The conductivity of EG reinforced LLDPE was measured using a two-point resistivity probe system with the minimum limit of  $10^{-8}$  S/cm. The hot-pressed sample was cut into specimens for testing of  $12 \times 8 \times 0.25$  mm<sup>3</sup>. The first two set of samples (0.5 and 5 wt% of EG) does not conduct in all the cycles. The specimen started to conduct when 10.7 wt% of EG is added to the LLDPE. Therefore, only three sets of samples (10.7, 16, 56 wt% EG) are analysed for the electrical testing.

Conduction in graphene-based composites is usually described in terms of conventional static percolation theory, which is adequate in describing the dynamic process of conductive network formation. When preparing polymer/graphene composites, a high sheer is often applied during mixing in order to disperse EG in polymer matrix, leaving them separated from each other, resulting in low composite conductivity. These well dispersed and separated EGs are however reported to aggregate in the melt given sufficient temperature and time leading to high orders of magnitude improvement in composite conductivity. This means that for the same concentration of EG, a composite can show very different levels of conductivity. For this reason, one should be careful in comparing data reporting conductivity and percolation threshold values. This phenomenon is been termed as *Dynamic Percolation*. For filler content greater than the percolation threshold, the percolation curve can be fitted by an exponential function (1),

$$\sigma = \sigma_o \left( p - p_c \right)^t,\tag{1}$$

where  $\sigma_o$  is the scaling factor,  $p_c$  is the percolation threshold, t is the characteristic exponent which depends on the dimensionality of the system (t ~ 2 for 3D system; t ~ 1.3 for 2D system).  $p_c$  is inversely proportional to the particle aspect ratio [31]. This is the reason why conductive nanoparticles like GNPs, CNTs are interesting as fillers.

Fig. 9 shows the variation of the electrical conductivity of LLDPE / 10.7 wt% EG nanocomposites as a function of the P&F cycles. Like most polymers, LLDPE is not electrically conductive and its room temperature electrical resistivity is  $3.3e24 - 3e25 \ \mu\Omega$ .cm. The addition of EG greatly improved its conductivity with a sharp transition from an electrical insulator to an electrical conductor. The

percolation threshold value of the conducting composite is about 10.7 wt% of expanded graphite, which is much higher than the other polymer composites. The percolation threshold for the electrical conductivity depends very much on the geometry of the conductive filler. Filler with a higher aspect ratio such as sheet-like or fibre-like filler, has an advantage in forming a conductive network in polymer matrix than that of filler in either round or ellipse shape [32].

Even though the composite has a high percolation threshold, it was found that the EG particles were increasingly distributed and dispersed in LLDPE with the number of cycles and mainly oriented on the plane of the samples. This find was confirmed by electrical resistivity measurements. A low in -plane resistivity (about 300  $\Omega$  cm, corresponding to a conductivity of 0.003 S/cm, from Fig. 9) was found for nanocomposites prepared between 150 to 500 cycles. For each sample, the resistivity out- of plane was about 10.000 times higher than the in–plane resistivity (Fig. 9).

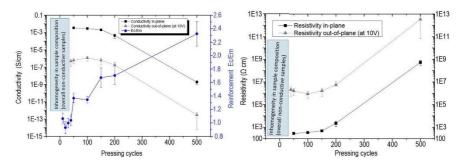


Figure 9. Electrical conductivity and resistivity of LLDPE / 10.7 wt% EG as a function of P&F cycles.

The conductivity data collected from the three different wt % of EG (10.7, 16 and 56 wt% at 200 cycles) are presented in Fig. 10 and it revealed that at lower filler concentration (below percolation threshold value of 10.7 %), the small EG particles that tend to aggregate into larger low aspect ratio clusters. It tends to disperse more evenly into the polymer matrix, thereby resulting in fewer particle-particle interactions at a given loading level and consequently leading to lower or no conductivity polymer composite. As the filler concentration is increased beyond the threshold concentration, the conductivity of all LLDPE composites is observed to increase rapidly.

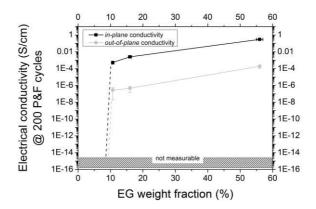


Figure 10. Electrical conductivity of LLDPE / EG composites at 200 cycles for different EG concentration.

Once the electrical percolation has been achieved, the increase in conductivity as a function of filler loading can be also modelled by a simple power-law expression (2),

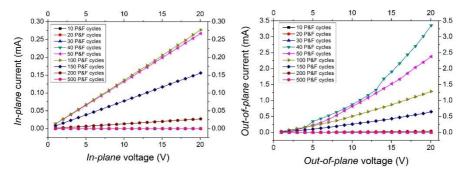
$$\sigma_c = \sigma_f (\phi - \phi_c)^t, \tag{2}$$

where  $\phi$  is the filler volume fraction,  $\phi_c$  is the percolation threshold,  $\sigma_f$  is the filler conductivity,  $\sigma_c$  is the composite conductivity and t is a scaling exponent [33]. The filler need not be in the direct contact for current flow; rather, conduction can take place via tunnelling between thin polymer layers surrounding the filler particles and this tunnelling resistance is the limiting factor in the composite conductivity [33]. Chen et al [34] reported that CNT /epoxy nanocomposites have an electrical percolation threshold as low as approximately 0.0025 wt%, which is by far the lowest, reported for any graphene-based nanocomposites. These exceptional results attribute to the kinetic percolation that most notably arises in composites with a low viscosity during processing. Eventually, it forms a three-dimensional network of filler content that electrically percolates at a much lower loading than possible with well dispersed and randomly oriented fillers. It has been said that a high degree of dispersion may not necessarily yield the lowest electrical percolation [33]. Indeed, the lowest percolation threshold achieved thus far for a graphene-based polymer nanocomposite (~ 0.15 wt% [32]) was observed when the filler was not homogeneously dispersed in the polymer matrix, but rather segregated from the matrix to form a conductive network.

Alignment of the filler also plays a major role in the onset of the electrical percolation. When the EG particles are aligned in the matrix, there are, at least at

relatively low concentrations (0.5 and 5 wt %), fewer contacts between them and thus the percolation threshold would increase till the composite starts to conduct at higher filler concentrations.

A series of SEM micrographs obtained from the samples of different filler concentration and different cycles are presented in the next section. The SEM images of the LLDPE/EG samples near the threshold concentration (10.7 wt%) reveal the existence of EG particles exhibiting short and long-range connectivity in the plane and through the thickness of the composite film. The SEM images from the LLDPE/10.7 wt% EG samples, show that the fibres are primarily, but not exclusively, aligned in the specimen plane throughout the thickness of the sample. Because of this difference in morphological behaviour of the sample, the electrical conductivity also changes its behaviour according to the orientation of the filler content. The in plane and the out of plane current for the LLDPE/10.7 wt% at different cycles as a function of applied voltage are shown in the Fig. 11.



*Figure 11. Variation of current vs voltage for in –plane and out-of-plane filler orientation* 

Dispersion of graphite is also important to the variation of percolation threshold for conductivity transition in the composites. In my experiment, I used long time, intensive pressing and folding (500 cycles) to promote fine dispersion for the composites. The sufficient adsorption of the LLDPE molecular chains onto various pores of the expanded graphite was also the likely factor contributing to good dispersion. If the solution concentration is too high, the higher viscosity may hinder the polymer chains from entering the minor pores of the graphite and thus lead to poor dispersion of graphite flakes in the polymer. Also, the variation of current vs voltage for in –plane and out-of-plane orientation for the filler content above the percolation threshold (i.e., 10.7, 16, 56 wt % EG) has been shown in the Fig. 12.

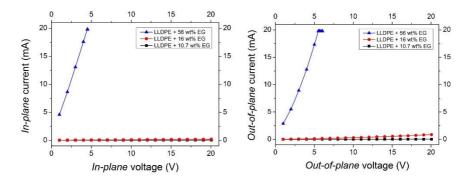


Figure 12. Variation of current vs voltage for in –plane and out-of-plane filler orientation for different filler concentration at 200cycles

### 3.3. Morphological Characterization

As the mechanical and the electrical property enhancements correlate strongly with nanocomposite microstructure, effective characterization of morphology is also important in establishing the structure-property relationships for these composites. SEM studies are perhaps the most common means by which the state of dispersion can be assessed. The expanded graphite used here is a particlebased filler (rather fibre-based filler), as it has the high aspect ratio. The higher aspect ratio platelets are generally found to be beneficial to the mechanical, electrical and thermal properties of the composite material [33]. An exfoliated morphology of EG is thus usually desired as it provides higher aspect ratio platelets relative to stacked or intercalated platelets. The cross-sectional analysis of the composite is also performed using SEM which has been used to evaluate the dispersion of EG as well as to examine the surface for pull -out, possibly giving insight into the strength of interfacial adhesion. Exfoliated graphene-based materials are often compliant and when dispersed in a polymer matrix are typically not observed as a rigid disk, but rather as bent or crumpled platelets as shown in Fig. 13. The cross section of the cryogenically broken sample for different filler content at 200 cycles is shown in the Fig. 14 a, b, c. The images show that the graphene is very well dispersed at the higher concentration and the three-dimensional network of the conductive fillers increase as the filler concentration increase. This leads to the higher percolation content at 10.7 wt%. The analogous images acquired from the LLDPE / EG samples (Fig. 14d) show that the fibres are primarily, but not exclusively, aligned in the specimen plane throughout the thickness of the sample.

P. K. Rajamani and R. Boros - Acta Technica Jaurinensis, Vol. 11, No. 4, pp. 185-205, 2018

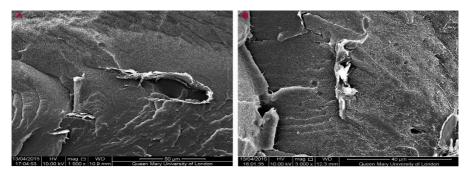


Figure 13. Identified graphite sheets in cryogenically broken sample of LLDPE / 0.5 wt% EG after A) 30 P&F cycles B) 200 P&F cycles

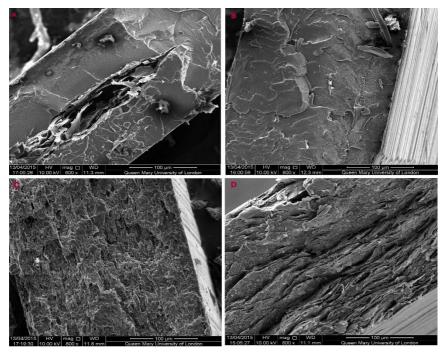


Figure 14. SEM images of cryogenically broken samples of 0.5 wt% EG content a)10 cycles b) 200 cycles c) 500 cycles and d) 56 EG wt% at 200 cycles

# 4. Conclusions

In this study, we focused on studying the mechanical and the electrical properties of LLDPE/EG nanocomposites with different filler content and different P&F cycles. Apparently, the EG system demonstrated better properties from a higher aspect ratio, as the filler content increases as well the pressing cycles increases. Some specific conclusions could be drawn as follows,

- The materials studied were fabricated in an alternative top-down production technique called Pressing & Folding technique and demonstrated the useful mechanical and thermal properties along with the morphological study of different filler content which was processed for different P&F cycles. Future development of graphite or graphene-based nanocomposites awaits better dispersion of the nano fillers in the polymer matrix and the control of percolation transition in applications.
- Viscosity increased with the addition of graphite fillers. The introduction of EG increased the stiffness of the composites. At 56 wt%, which is considered as the maximum filler loading content in the polymer showed high viscosity than the lower EG content. The difference was attributed to EG's higher area to volume and aspect ratios which promoted a more viscous flow in LLDPE matrix.
- Electrical conductivity for the composites showed a transition from an insulator to a conductor. But it requires a minimum of 10.7 wt% of EG content to reach the percolation threshold. It was also found that the EG particles were increasingly distributed and dispersed in LLDPE with the number of cycles and mainly oriented on the plane of the samples. Although LLDPE is a good insulator, a low in-plane resistivity was found for nanocomposites prepared with more P & F cycles (200 cycles). For each sample, the out of -plane resistivity was about 10.000 times higher than the in-plane resistivity.
- The results from the micro-structures of the prepared samples were studied by analysing the cross sections of the cryogenically broken and mechanically broken (tensile) samples by SEM. It was found that most of the EG particles are mainly oriented on the plane of the samples.
- Both tensile and electrical measurements indicated EG were better filler in the LLDPE. Although overall improvement in mechanical properties was not impressive, but our results clearly confirmed the advantages of using EG by P&F method to enhance both the electrical conductivity and mechanical strength and stiffness of LLDPE. Therefore, the role of EG as a reinforcement phase was unambiguously established.

# Acknowledgement

I wish to specially thank the Nanovision and Nanoforce Laboratory at Queen Mary University of London for their technical assistance and valuable support in terms of materials and equipment.

# References

- S. Park, R.S. Ruoff, Chemical methods for the production of graphenes, Nature Nanotechnology 4 (4) (2009) pp. 217-224.
   DOI: <u>https://doi.org/10.1038/nnano.2009.58</u>
- [2] A.K. Geim, K.S. Novoselov, The rise of graphene, Nature Materials 6 (3) (2007) pp. 183-191.
   DOI: <u>https://doi.org/10.1038/nmat1849</u>
- [3] H. Shioyama, Cleavage of graphite to graphene, Journal of Materials Science Letters 20 (6) (2001) pp. 499-500.
   DOI: <u>https://doi.org/10.1023/A:1010907928709</u>
- [4] Y. Li, S. Wang, Q. Wang, M. Xing, A comparison study on mechanical properties of polymer composites reinforced by carbon nanotubes and graphene sheet, Composites Part B 133 (2018) pp. 35-41. DOI: <u>https://doi.org/10.1016/j.compositesb.2017.09.024</u>
- [5] V.D. Punetha, S. Rana, H.J. Yoo, A. Chaurasia, J.T. McLeskey Jr, M.S. Ramasamy, N.G. Sahoo, J.W. Cho, Functionalization of carbon nanomaterials for advanced polymer nanocomposites: A comparison study between CNT and graphene, Progress in Polymer Science 67 (2017) pp. 1-47. DOI: https://doi.org/10.1016/j.progpolymsci.2016.12.010

DOI. <u>mtps.//doi.org/10.1010/j.progporymsci.2010.12.010</u>

 [6] H.Yao, H.J. Sue, Preparation of epoxy nanocomposites containing welldispersed graphene nanosheets, Composites Science and Technology 146 (2017) pp. 161-168.
 DOI: <u>https://doi.org/10.1016/j.compscitech.2017.04.026</u> P. K. Rajamani and R. Boros - Acta Technica Jaurinensis, Vol. 11, No. 4, pp. 185-205, 2018

- J.Z. Liang, Q. Du, G.C. Tsui, C.Y. Tang, Tensile properties of graphene nano-platelets reinforced polypropylene composites, Composites Part B 95 (2016) pp. 166-171.
   DOI: https://doi.org/10.1016/j.compositesb.2016.04.011
- [8] Y. Guo, G. Xu, X. Yang, K. Ruan, T. Ma, Q. Zhang, J. Gu, Y. Wu, H. Liu, Z. Guo, Significantly enhanced and precisely modelled thermal conductivity in polyimide nanocomposites with chemically modified graphene via in situ polymerization and electrospinning-hot press technology, Journal of Materials Chemistry C 6 (12) (2018) pp. 3004-3015. DOI: <u>10.1039/C8TC00452H</u>
- Y. Lan, Z. Guo, Electrically conductive thermoplastic polyurethane/ polypropylene nanocomposites with selectively distributed graphene, Polymer 97 (2016) pp. 11-19.
   DOI: <u>https://doi.org/10.1016/j.polymer.2016.05.017</u>
- Y. Cui, S. Kumar, Gas barrier performance of graphene/polymer nanocomposites, Carbon 98 (2016) pp. 313-333.
   DOI: <u>https://doi.org/10.1016/j.carbon.2015.11.018</u>
- A.P. Bafana, E.K. Wujcik, Polypropylene nanocomposites reinforced with low weight percent graphene nanoplatelets, Composites Part B: Engineering 109 (2017) pp. 101-107.
   DOI: <u>https://doi.org/10.1016/j.compositesb.2016.10.048</u>
- J.R. Potts, R.S. Ruoff, Graphene-based polymer nanocomposites. Polymer 52 (1) (2011) pp. 5-25.
   DOI: <u>https://doi.org/10.1016/j.polymer.2010.11.042</u>
- B.Z. Jang, A. Zhamu, Processing of nanographene platelets (NGPs) and NGP nanocomposites: A review, Journal of Materials Science 43 (15) (2008) pp. 5092-5101.
   DOI: https://doi.org/10.1007/s10853-008-2755-2
- K. Kalaitzidou, H. Fukushima, L.T. Drzal, A new compounding method for exfoliated graphite-polypropylene nanocomposites with enhanced flexural properties and lower percolation threshold, Composites Science and Technology 67 (2007) pp. 2045-2051.
   DOI: <u>https://doi.org/10.1016/j.compscitech.2006.11.014</u>

P. K. Rajamani and R. Boros - Acta Technica Jaurinensis, Vol. 11, No. 4, pp. 185-205, 2018

- [15] W.S. Kang, S.J. Park, Thermal, impact and toughness behaviours of expanded graphite/graphite oxide-filled epoxy composites, Composites Part B: Engineering 94 (2016) pp. 238-244.
   DOI: <u>https://doi.org/10.1016/j.compositesb.2016.03.052</u>
- G. Wang, Z.Dai, L. Liu, H.Hu, Q. Dai, Z.Zhang, Tuning the interfacial mechanical behaviours of monolayer graphene/PMMA nanocomposites, ACS applied materials & Interfaces 8 (2016) pp. 22554-22562.
   DOI: <u>10.1021/acsami.6b03069</u>
- [17] K.Wu, Q.Fu, Largely enhanced thermal and electrical conductivity via constructing double percolated filler network in polypropylene/expanded graphite – Multi-wall carbon nanotubes ternary composites, Composites Science and Technology 130 (2016) pp. 28-35. DOI: <u>https://doi.org/10.1016/j.compscitech.2016.04.034</u>
- [18] K. Gaska, X.Xu, S. Gubanski, R. Kadar, Electrical, Mechanical, and Thermal Properties of LDPE Graphene Nanoplatelets Composites Produced by Means of Melt Extrusion Process. Polymers 9 (1) (2017) pp. 11-23. DOI: <u>https://doi.org/10.3390/polym9010011</u>
- [19] A.A. Vasileiou, M. Kontopoulou, A.Docoslis, A noncovalent compatibilization approach to improve the filler dispersion and properties of polyethylene/graphene composites, ACS Applied Material Interfaces 6 (3) (2014) pp. 1916-1925.
   DOI: 10.1021/am404979g
- H.R. Mamagani, V. Haddadi-Asl, K. Khezri, M.S. Kalajahi, Polystyrenegrafted graphene nanoplatelets with various graft densities by atom transfer radical polymerization from the edge carboxyl groups, RSC Advances 4 (47) (2014) pp. 24439-24452.
   DOI: <u>http://dx.doi.org/10.1039/c4ra03451a</u>
- S. Roy, X. Tang, T. Das, L. Zhang, Y. Li, S. Ting, X. Hu, C.Y. Yue, Enhanced Molecular Level Dispersion and Interface Bonding at Low Loading of Modified Graphene Oxide To Fabricate Super Nylon 12 Composites, ACS Applied Materials Interfaces 7 (5) (2015) pp. 3142-3151. DOI: <u>10.1021/am5074408</u>

P. K. Rajamani and R. Boros – Acta Technica Jaurinensis, Vol. 11, No. 4, pp. 185-205, 2018

- [22] H.Xu, L.X. Gong, X. Wang, L. Zhao, Y.B. Pei, G. Wang, Y.J. Liu, L.B. Wu, J.X. Jiang, L.C. Tang, Influence of processing conditions on dispersion, electrical and mechanical properties of graphene filled silicone rubber composies, Composites Part A: Applied Science and Manufacturing 91 (1) (2016) pp. 53-64. DOI: <u>https://doi.org/10.1016/j.compositesa.2016.09.011</u>
- [23] T. Kuilla, S. Bhadra, D. Yao, N.H. Kim, S. Bose, J.H. Lee, Recent advances in graphene based polymer composites, Progress in Polymer Science 35 (11) (2010) pp. 1350-1375. DOI: https://doi.org/10.1016/j.progpolymsci.2010.07.005
- [24] O.M. Istrate, K.R. Paton, U. Khan, A. O'Neil, A.P. Bell, J.N. Coleman, Reinforcement in melt-processed polymer-graphene composites at extremely low graphene loading level, Carbon 78 (2014) pp. 243-249. DOI: https://doi.org/10.1016/j.carbon.2014.06.077
- [25] L.C. Tang, Y.J. Wan, D. Yan, Y.B. Pei, L. Zhao, Y.B. Li, L.B. Wu, J.X. jiang, G.Q. Lai, The effect of graphene dispersion on the mechanical properties of graphene/epoxy composites, Carbon 60 (2013) pp. 16-27. DOI: https://doi.org/10.1016/j.carbon.2013.03.050
- [26] X. Zhang, X.Fan, H. Li, C. Yan, Facile preparation route for graphene oxide reinforced polyamide 6 composites via in situ anionic ring-opening polymerization, Journal of Materials Chemistry 22 (45) (2012) pp. 24081-24091. DOI: 10.1039/C2JM34243J
- [27] H. Wang, Q. Hao, X. Yang, L. Lu, X. Wang, Effect of Graphene Oxide on the Properties of Its Composite with Polyaniline, ACS Applied Materials Interfaces 2 (3) (2010) pp. 821-828. DOI: http://dx.doi.org/10.1021/am900815k
- H. Hu, G. Zhang, L. Xiao, H. Wang, Q. Zhang, Z. Zhao, Preparation and [28] electrical conductivity of graphene/ultrahigh molecular weight polyethylene composites with a segregated structure, Carbon 50 (12) (2012) pp. 4596-4599.

DOI: https://doi.org/10.1016/j.carbon.2012.05.045

P. K. Rajamani and R. Boros - Acta Technica Jaurinensis, Vol. 11, No. 4, pp. 185-205, 2018

- [29] L. Hu, J. Tu, S. Jiao, J. Hou, H. Zhu and D. J. Fray, In situ electrochemical polymerization of a nanorod-PANI–Graphene composite in a reverse micelle electrolyte and its application in a supercapacitor, Physical Chemistry Chemical Physics 14 (45) (2012) pp. 15652-15656. DOI: <u>http://dx.doi.org/10.1039/c2cp42192e</u>
- [30] W. Zheng, X. Lu, S.C. Wong, Electrical and mechanical properties of expanded graphite-reinforced high-density polyethylene, Journal of Applied Polymer Science 91 (5) (2004) pp. 2781-2788.
   DOI: <u>https://doi.org/10.1002/app.13460</u>
- [31] D. Stauffer, A. Aharony, Introduction of Percolation Theory, 2<sup>nd</sup> Edition, Taylor and Francis, London, 1985.
- [32] Y.S. Wang, M.A. O'Gurkis, J.T. Lindt, Electrical properties of exfoliatedgraphite filled polyethylene composites, Polymer Composites 7 (5) (1986) pp. 349-354.
   DOI: https://doi.org/10.1002/pc.750070512
- [33] M. Zhang, Y. Li, Z. Su, G. Wei, Recent advances in the synthesis and applications of graphene–polymer nanocomposites, Polymer Chemistry 6 (34) (2015) pp. 6107-6124.
   DOI: <u>https://doi.org/10.1039/C5PY00777A</u>
- [34] X.M. Chen, J.W. Shen, W.Y. Huang, Novel electrically conductive polypropylene/graphite nanocomposites, Journal of Materials Science Letters 21 (3) (2002) pp. 213-214.
   DOI: <u>http://dx.doi.org/10.1023/A:1014708808230</u>



Vol. 11, No. 4, pp. 206-217, 2018 DOI: 10.14513/actatechjaur.v11.n4.480 Available online at acta.sze.hu

# Pinpoint Loading Examinations of Poly(lactic acid) Biopolymers

## L. Kotrocz, P. Bakonyi

#### Budapest University of Technology and Economics, Department of Polymer Engineering Muegyetem rkp. 3., 1111Budapest, Hungary e-mail: bakonyi@pt.bme.hu

Abstract: We have made a new mounting unit for Dynamic Mechanical Analysis (DMA) equipment, suitable for measuring microhardness and indentation, and examined its usability on thermoplastic poly(lactic acid) (PLA) material. We used a Vickers-type microhardness indenter, and examined the dimensions of the indented area and the loading force. This novel unit is suitable for measuring the short and long-term behavior of the material using pinpoint load not only at room temperature (20-23 °C) – like common hardness tests – but at a wide range of temperatures.

Keywords: poly(lactic acid); biopolymer; pinpoint load; Vickers indentation; Dynamic Mechanical Analysis

## **1. Introduction**

As technology develops, polymers are more and more widely used. In some cases, they can replace metals, due to their adequate strength, low density, good corrosion resistance and vibration damping ability, but they can behave very differently compared to metals. In the case of polymers it is not enough to examine quasi-static mechanical characteristics (tensile and flexural tests) because they react to long-term loading with continuously increasing deformation; this is called creeping. Therefore, it is an important rule for designers that they should design parts not for lifetime but for maximal possible deformation. Creep is most widely determined with tensile and flexural tests, but torsion and compression loading is also used. Compression loading can be area or pinpoint loading. In this paper we studied the latter one [1].

Nowadays, with the advancement of the environment-conscious approach, more and more research is directed at the recycling of plastics and the development of biopolymers. Biopolymers can replace petroleum-based polymers in many cases. Perhaps the most well-known of these is Poly(lactic acid) (PLA). It is used in many areas, such as packaging, foams, textiles (geotextiles, towels) and even medical applications. PLA is mechanically excellent with an elastic modulus of 3000–4000 MPa and a tensile strength of 50–70 MPa, but it's brittle; its tensile elongation is around 3-5% and its impact strength is low. It can be processed with conventional technologies. PLA composites and nanocomposites are used more and more. Fiber or nanoscale fillers can modify the different properties of PLA [2-6].

Pinpoint compression loading can be examined very well with a hardness test. The hardness of materials is an important characteristic that helps designers select the right material. It is defined as the resistance of a solid material to penetration by a body. Depth-sensitive hardness testing (depth-sensing indentation – DSI) was developed while the advantages of hardness testing were retained. During this process, the measuring body is pushed into the surface at constant speed or with a constant force, and then removed. The geometry of the impression depends on the depth of indentation, which in turn depends on the loading force, and the time of loading, and of course, environmental parameters, such as humidity and temperature. Various characteristics can be determined from it, such as dynamic hardness, conventional hardness, modulus of elasticity, and elastoplastic mechanical properties [7-8].

The advantage of depth-sensing indentation is that measurements can be made in the millimeter, micrometer and nanometer range as well. This facilitates the testing of various thin films, or new materials produced by nanotechnology (e.g. nanocomposites), even with very little loading forces (in the case of nanohardness testing, it can be 0.01 mN). This procedure may be suitable for the testing of products made of PLA [4-5]. A disadvantage is that the measurement results are always local, and can be very different. Also, there are two important phenomena to be mentioned. Next to the indenter, at the side of the indented area, the surface can sink (sinkingin), or can be raised because material is pushed out (pilling-up), and these can cause inaccuracies (Fig. 1). For this reason, several tests have to be performed for accurate results [7].

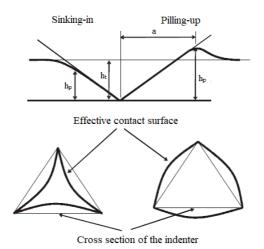


Figure 1. Sinking-in and pilling-up [2]

According to the literature we analyzed, [9-11] this procedure, combined with creep tests, is used successfully on polymer materials. The indenters were mostly Vickers- or Berkovich-type indenters. Usually, finite element analysis is performed as well, to support the measurement results.

The literature [9-11] shows that although this method is spreading, tests are performed almost exclusively at room temperature. However, polymer objects can behave differently at other temperatures and humidity; therefore we decided that tests should be performed in a wide range of temperatures. In order to provide the small loading forces and constant temperature required for the test, we designed a mounting unit suitable for pinpoint loading. In a dynamic mechanical analyzer (DMA), short, long and also cyclical tests can be performed with the unit.

#### 2. Methods and Materials

#### 2.1. The mounting unit designed

We designed the mounting unit for a TA Instruments (USA) Q800 device. The analyzer can perform various tests (tensile, compression, flexural and shear tests) in a wide temperature range (-145 °C to +600 °C), and it has a frequency range of 0.01 Hz to 200 Hz. Both constant and periodic loading can be applied up to 18 N. The device can determine the mechanical and viscoelastic properties of various materials [12].

We designed the mounting unit based on an existing fixture used for compression tests. We had two concepts, one where the indenter is fastened in the middle of the fixture, and another called the "horse race track" design (this allows the positioning of the indenter). Fig. 2 shows the two designs.

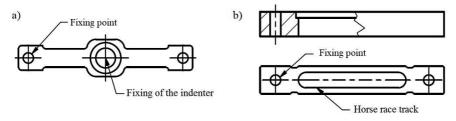
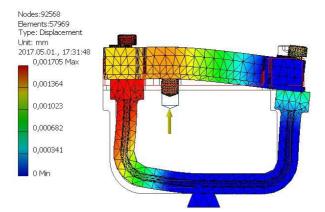


Figure 2. Designs of the fixture, fixed (a) and "horse race track" (b) design

We tested the two concepts with finite element analysis at room temperature to see what deformations occur. Fig. 3 shows that in the case of the horse race track design, in an extreme position, at a load of 18 N, the frame deforms as well, which can damage the air bearing system of the DMA and the mounting unit itself, especially if deformation resulting from temperature change is also taken into account, because at higher temperatures the deformations can be bigger. The asymmetric load caused by the indenter in the extreme position can occasion buckling of the 150-200 mm long and ¼ inch diameter spindle supported by the air bearing. The spindle is fixed at its lower end and extends beyond the air bearing about 80-100 mm, therefore the deformation of the whole system is more than what is shown. Therefore, we selected as the final design, the centrally positioned, gripped indenter design to avoid asymmetric loading.



*Figure 3. The finite element simulation results of the horse race track in an extreme position* 

We made the sample holder so that the sample can be positioned. This makes it possible to perform several measurements on a single sample. Thanks to the vice design, the specimen can be tightly gripped and so it does not move during the test. The final design manufactured from ST32 stainless steel can be seen in Fig. 4.

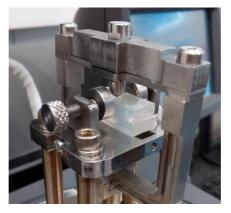


Figure 4. The designed fixture and the gripped PLA specimen

#### 2.2. The test program and the investigated material

We tested 3100 HP from NatureWorks (Minnetonka, MN, USA) PLA, whose D-lactide content (0.5%) was provided by the manufacturer. The granules were dried at 80 °C for 6 hours prior to processing to remove residual moisture and thus to avoid

hydrolitical degradation during processing. ISO 527-2 type 1A standard dumbbellshaped specimens were injection molded with an Arburg Allrounder 370S 700-290 injection molding machine (Lossburg, Germany) equipped with a 30 mm diameter, L/D = 25 screw. Injection rate was 50 cm<sup>3</sup>/s, holding pressure was 600 bars, holding time was 20 sec, residual cooling time was 40 sec, and melt and mold temperature were 190 °C and 25 °C, respectively.

The specimens were cut out from the injection molded dumbbells and their dimensions were 10 mm x 20 mm x 4 mm. We performed the test on the 10 mm x 20 mm surface, which was the polished surface of the mold. We increased loading force (F [N]) from 3 N to 15 N, by 3 N in each step. The loading time was 30 s, 60 s and 90 s. The temperature was 35 °C in every case. In the tests, we used a Vickers microhardness indenter, which is pyramid-shaped and is made of diamond. This geometry reduces dependence on loading force. The parameters measured on the negative mark of the indenter can be seen in Fig. 5, where *a* and *b* are the side length,  $d_1$  and  $d_2$  are the diagonals and *h* is the cone height of the Vickers intender.

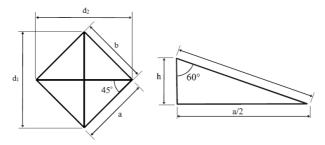


Figure 5. The impression of a Vickers indenter and its geometrical parameters [7]

The testing program consisted of the following steps: we heated the heat chamber to 35 °C, and held this temperature for 3 minutes. Then we applied the loading force. After loading ceased, we analysed the indented area with an optical microscope (Olympus BX51M, Japan), and measured the diagonals of the indented areas ( $d_1$ ,  $d_2$  [µm]).

#### 3. Results and discussion

As mentioned above, evaluation started with the microscopy examination of the indented areas. We experienced pilling-up and sinking in several cases during the tests; an example is shown in Fig 6.

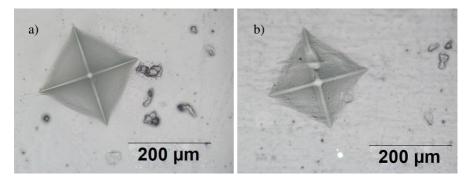


Figure 6. Pilling-up (a) and sinking-in (b) phenomena on the PLA material

We measured the diagonals of the impressions. The diagonal length–force curve for the 30-second loading time test is displayed in Fig. 7.

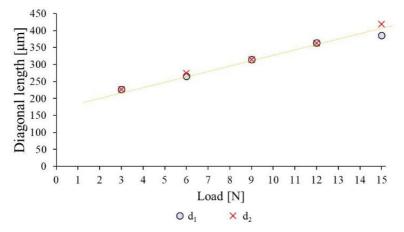


Figure 7. The diagonals as a function of loading force, in the case of 30 s loading time

It is visible that the points are on one line with little deviation. This proves that the impressions are symmetrical; therefore, the fixture can be used in indentation tests in a DMA. Because of the symmetry, the diagonals can be averaged. In Fig. 8. we displayed the averaged diagonals as a function of loading, in the case of different loading times.

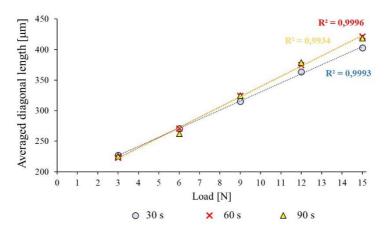


Figure 8. Averaged diagonal length as function of loading force

If the diagonals are known, the projected area ( $A \ [\mu m^2]$ ) of the pyramid created by the indenter can be calculated with equation (1), and its change can be plotted as a function of the force (Fig. 9).

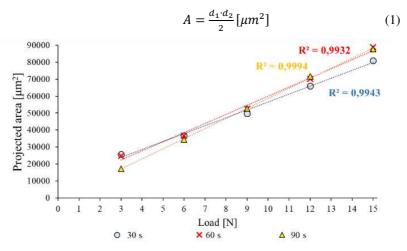


Figure 9. Projected area as a function of loading force

From the measured diagonals, the depth of the impression can be calculated with equation (2):

$$h = \frac{\mathrm{d}}{2 \cdot \sqrt{2} \cdot \tan\left(\frac{\theta}{2}\right)},\tag{2}$$

where  $h \ [\mu m]$  is the depth of the indent,  $d \ [\mu m]$  is the average length of the diagonal and  $\theta \ [rad]$  is the angle of the indenter (136°). Its change can also be displayed as a function of the force (Fig. 10.).

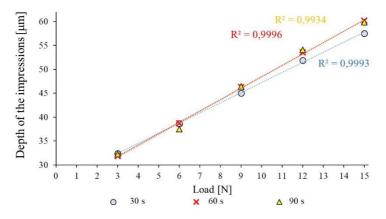


Figure 10. Depth of the impression as a function of loading force (enlarged)

With the method of least squares, we can align linear trend lines for the points in each case. The lines all have a determination coefficient above 99%. All of the diagrams demonstrate that longer loading times result in deeper and bigger impressions.

The diagrams show that larger force resulted in larger impressions. Also, if loading time was longer, it also resulted in larger impressions. There is no significant difference between 60 s and 90 s results at this temperature. Therefore, in the future, it is enough to do one of the measurements, or study that at another temperature.

The diagonal sizes were measured as soon as possible (within 30 seconds), but it should be noted that the time and temperature dependence of diagonal and depth recovery requires additional tests, because there is instantaneous recovery due to the momentary elastic deformation component, which is typical for polymers, along with delayed elastic and irreversible components [13].

From the calculated results, in the future, we can determine the hardness of the material, and also other mechanical properties, such as elastic modulus.

# 4. Conclusions

The tests showed that the fixture we designed is suitable for pinpoint loading tests with DMA equipment. This method can be used for the mechanical testing of the surface of polymer products. We can calculate the projected area and the depth of the impressions, which we can use to determine the hardness of the measured material. Also, in the future, we can compare the data calculated from the optical microscopy images with the indentation depths measured with the DMA device. Therefore, we will be able to examine the phenomenon of creep, and the impressions need not be measured with an optical microscope.

Thanks to the DMA equipment, the procedure can be used not only for static tests but also dynamic and cyclic tests. In addition, tests can be performed in a wide range of temperatures. In the case of longer tests, creep has to be expected, which is characteristic to polymers. In smaller sizes, creep can be more pronounced; therefore more detailed tests need to be done regarding this.

# Acknowledgements

The project is funded by the National Research, Development and Innovation (NKFIH) Fund, Project title: "Developing a new generation of customized medical implants and medical aids for additive technologies"; The application ID number: NVKP\_16-1-2016-0022. The developers are grateful for the support.

# References

- J.S. Urzumtsev, R.D. Maksimov, Deformation of plastics. Prognostics on the basis of similarity principles, 1st edition, Műszaki Könyvkiadó, Budapest, 1982.
- [2] Poly(lactic acid): Synthesis, Structures, Properties, Processing, and Applications, in: R. Auras, L. T. Lim, S. E. M. Selke, H. Tsuji (Eds), 1st Edition, John Wiley & Sons, Inc., Hoboken, New Jersey, 2010. doi: <u>10.1002/9780470649848</u>
- [3] S. Shankar, J.W. Rhim, Polymer Nanocomposites for food packaging Applications, in: A. Dasari, J. Njuguna (Eds), Functional and Physical Properties of Polymer Nanocomposites, John Wiley & Sons, Inc., Hoboken, New Jersey, 2016, pp. 29-55. doi: 10.1002/9781118542316.ch3

- [4] S. C. Cifuentes, E. Frutos, R. Benavente, V. Lorenzo, J. L. González-Carrasco, Assessment of mechanical behavior of PLA composites reinforced with Mg miceo-particles through depth-sensing indentations analysis, Journal of the mechanical behavior of biomedical materials, 65 (2017) pp. 781-790. doi: <u>10.1016/j.jmbbm.2016.09.013</u>
- [5] S.C. Cifuentes, E. Frutos, R. Benavente, J.L. González-Carrasco, V. Lorenzo, Strain rate effect on semi-crystalline PLLA mechanical properties measured by instrumented indentation tests, 59 (2014) pp. 239-246. doi: <u>10.1016/j.eurpolymj.2014.07.034</u>
- [6] S. Hajba, T. Tábi, Investigation of long cellulose fibre reinforced and injection moulded poly(lactic acid) biocomposites, Acta Technica Jauriensis, 11 (3) (2018) pp. 150-164. doi: <u>10.14513/actatechjaur.v11.n3.469</u>
- [7] P. M. Nagy, Investigation of block and surface mechanical properties of structural polymers by indentation measurement technologies, PhD thesis, Eötvös Lóránd University (2007).
- [8] J. Gubicza, A. Juhász, J. Lendvai, A new method for hardness determination from depth sensing indentation test, Journal of Materials Research, 11 (12) (1996) 2964-2967. doi: 10.1557/JMR.1996.0376
- [9] P. J. Wei, W. X. Shen, J. F. Lin, Analysis and modeling for time-dependent behavior of polymers exhibited in nanoindentation tests, Journal of Non-Crystalline Solids, 354 (33) (2008) pp. 3911–3918. doi: <u>10.1016/j.jnoncrysol.2008.05.016</u>
- [10] R. Seltzer, Y.-W. Mai, Depth sensing indentation of linear viscoelastic-plastic solids: A simple method to determine creep compliance, Engineering Fracture Mechanics, 75 (17) (2008) pp. 4852-4862. doi: 10.1016/j.engfracmech.2008.06.012
- K. Altaf, I. A. Ashcroft, R. Hague, Modelling the effect of moisture on the depth sensing indentation response of a stereolithography polymer, Computational Materials Science, 52 (1) (2012) pp. 112-117. doi: <u>10.1016/j.commatsci.2011.01.051</u>

- [12] TA Instruments, DMA Q800 user manual (2011) [cited 2017-10-12]. URL <u>http://www.tainstruments.com/wp-content/uploads/dma.pdf</u>
- [13] R. J. Crawford: Microhardness testing of plastics, Polymer Testing, 3 (1) (1982) pp. 37-42.
   doi: 10.1016/0142-9418(82)90011-3



# Halal Meat Supply Chain Traceability Based on HACCP, Blockchain and Internet of Things

# A. Rejeb

#### Széchenyi István University, Department of Logistics and Forwarding Egyetem tér 1, 9026 Győr, Hungary e-mail: abderrahmen.rejeb@gmail.com

- Abstract: Recently, Halal food has drawn remarkable attention of many consumers around the world. Besides to being unsafe, Halal food such as meat can encounter several issues throughout its supply chain and logistics. At any time, Halal integrity is not guaranteed and risks of becoming non-Halal is the major concern of all parties along the supply chain. To respond to Muslim consumers' trust concerns in Halal food, many traceability systems were proposed in previous studies based on emerging technologies and recommended to be incorporated into Halal food supply chains. Nevertheless, all of these systems are centralized, opaque and not enough transparent. To mitigate these problems, blockchain technology is introduced as a ground-breaking innovation with greater decentralization, visibility and transparency. This paper makes a major contribution in suggesting Halal meat supply chain traceability system for real-time food tracing based on embedding Islamic dietary law into HACCP, blockchain and Internet of Things.
- Keywords: Halal Meat Supply Chain; HACCP; Blockchain; Internet of Things; Traceability

## 1. Introduction

Generally, food supply chains are highly characterized by their complexity. In fact, products associated with food supply chains are scattered at each stage of production and between vast networks of different concealed actors for both supplier and consumer. This raises the question of managing effectively food supply chains as there are a huge concern and upsurge for not only food safety but for ensuring food compliance to certain religious restrictions as well. In fact, different religious groups are restrained to their own food restrictions and pay influential roles to prevent their followers from certain foods [1] such as Jews for Kosher, the prohibition of beef-

eating under Hinduism, and the Islamic Dietary Laws [2]. Besides, the consumers' decision to purchase food is not only guided by the perception of healthiness, safety and sensory traits (i.e., color, tenderness, flavor, and aroma) but by the strict compliance to religion [3]. In this respect, the Muslims' viewpoint to purchase some kind of food depends exclusively on its Halal status. Similarly, food turns from being sustenance to an integral dimension of culture and identity which impacts greatly supply chains and logistics.

Consumers' awareness and education of Halal foods grow together with a noticeable increase in the demand for Halal products on a global scale. Today, Halal foods are no longer considered as religious requirements for Muslims but non-Muslims are also involved in demanding and supplying this particular food group due to many reasons [4]. Moreover, aside from the response to the increasing pace of Muslim population, Halal products, especially food products account for a staggering US\$600 billion to US\$2.1 trillion [5]. This is consistent with [6] [7] [8] who claimed that non-Muslim consumer segment perceives Halal foods beyond the limit of religion. According to [6] [9] Halal status is a global symbol for food safety, quality insurance, and lifestyle choice. Furthermore, the Halal food industry has been globalized due to tourism and migration [10]. Plus, previous studies have demonstrated that Muslims are resistant to acculturation and attached to their ethos and religious dietary laws. Simply stated, they still eat Halal even after having immigrated to Non-Muslim countries. A study carried out by Hussaini showed that 75% of Muslims in the US still maintain their Islamic eating rules [11].

The increasing popularity of Halal food industry is not only attributable to the additional and value-added features of being safe, wholesome, hygienic and contamination-free principles during food production [12]. It is due to the market attractiveness and involvement of non-Muslim countries in supplying food products to Muslims. Argentina, Australia, Brazil, Canada, New Zealand, the United Kingdom and the United States are the major food producers and suppliers of many Muslim countries [13]. Hence, the globalized nature of Halal supply chains opens up windows of chances for many actors to join the lucrative industry. Billions of customers are targeted by Halal food companies and governments across the world are recognizing the potential of the Halal industry through the continuous support and promotion. This is manifested also by the establishment of Halal certification authorities, the material incentives for funding research on Halal related studies through universities and research centers as well as the offer of Halal training for industry practitioners [14]. Besides, Halal food industry leads to positive externalities in the development of Halal traceability and tracking systems to strengthen food industry and to alleviate all the probable risks and concerns.

There are many ethical and social questionable activities that transcend the layers of the food supply chain. In this vein, retailers in the food industry are facing challenges of foodborne diseases and are struggling to figure out the source of defective ingredients and to which stores they were delivered [15]. Also, consumers' confidence in the food industry was heavily decoyed after consecutive food safety scandals and frauds, namely mad cow disease, genetically modified foods and the European horse meat scandal in 2013. Not only that, Halal food industry may encounter several issues related to Halal integrity which could be questionable and even breached throughout the phases of supply chain and thus the product will be no longer fit for consumption by Muslim consumers. Several problems may put Halal products into the integrity question. From raw material until it reaches consumers. Halal food may encounter the risk of cross-contamination with Haram products, the non-compliance to Halal slaughtering and ill-intentions. Therefore, there is a problem of distrust to Halal supply chain actors. In response to confidence in Halal food products, Halal logo or certification on the product packaging is presented as the best way to inform the consumers about the compliance of food products to the Halal guidelines and Sharia law principles. Halal logos become very popular to allow customers to crosscheck Halal integrity of the product and support the purchasing decision for both Muslims and Non-Muslims [14]. However, possibilities of misleading consumers through fake logo or certificates are still present. This is because there are various Halal authorities or agencies in some Muslim and non-Muslim countries which resulted in multiple Halal standards and doubtful Halal certifications [16].

To mitigate the risks of Halal meat supply chain, a level of collaboration between the different partners of the chain should be maintained to ensure the trustworthiness of Halal food integrity. This implies to say that a trustful platform based on exchanging information, mutuality, and transparency entails mutual benefits across the whole Halal chain. In a similar way, many technologies are applied to Halal supply chain traceability and visibility in tandem with responding to these concerns. In their studies, Tan et al., Bahrudin et al., and Anir et al. emphasized strongly the adoption of information and communication technology (ICT) for Halal transportation such as Global Positioning System (GPS), Radio Frequency Identification (RFID), and Internet of Things (IoT) to monitor Halal logistics activities. Since the adoption of technology into Halal logistics and supply chain has gained recognition [14], the scope of this study is to propose a supply chain traceability system for Halal meat based on embedding Islamic dietary law into Hazard Analysis and Critical Control Points (HACCP) approach for application to Halal production and processing, Blockchain (BC) and Internet of Things (IoT).

Notwithstanding the potential of the novel BC technology to leverage the use of tracking systems such as RFID technology, this research topic remains overlooked

and unaddressed by academic researchers. BC is an emerging technology presented to ensure the storage of all information related to the food products in a shared, visible and transparent system for all the members along the supply chain [15]. Several BC projects have been implemented to track and trace the movements of food products throughout the stages of the supply chain and consequently to ensure the integrity of food products.

This paper will discuss Halal meat supply chain. Hence, it begins with a review of the concept of Halal and the relevant issues pertaining to the supply chain and logistics of Halal meat. The next section will present the typical design of Halal meat supply chain along with a process flow and relevant Halal critical points are identified with an example of HACCP management. The following section will introduce the details of the BC-based system with a discussion about the critical role of this technology to enforce customers' trust in Halal meat supply chain. The last section covers a brief conclusion for the paper.

#### 2. Literature Review

Halal is an Arabic word and it refers to the Islamic belief. Halal means all things and actions permitted by the Sharia (i.e., Islamic law). Similarly, Halal is an object or an action which is allowed or lawful to be used or taken according to the Islamic law [17]. The antonym of Halal is Haram which means prohibited or forbidden or unlawful. The ideas of Halal and Haram are clearly defined within the Islamic teachings and it is very important to understand that the scope of Halal is so wide since it is a way of life and embraces good conduct and righteous deeds [18]. Besides, Halal status extends also to lifestyle (such as clothing) and services (Islamic finance, hospitality, and logistics, etc) [19]. In the context of food, the Islamic law demonstrates that it is mandatory and crucial for Muslims to consume only Halal (permitted) food and to abstain from any activities that are or will lead to Haram. It is extensively mentioned in the Quran (The holy book) and the Sunnah the guidelines for Halal food:

"Forbidden to you is that which dies of itself, and blood, and the flesh of swine, and that on which any other name than that of Allah has been invoked, and the strangled (animal) and that beaten to death, and that killed by a fall and that killed by being smitten with the horn, and that which wild beasts have eaten, except what you slaughter, and what is sacrificed on stones set up (for idols) and that you divide by the arrows; that is a transgression." (The Quran: Al-Maedah 5:3)

Simply stated, Halal food should be acceptable (i.e., Sharia-compliant) and of good quality, wholesome and safe. Alcohol, pork, blood, dead meat, and meat which has not been slaughtered according to Islamic rulings are all considered Haram [20].

With respect to meat, Halal status is not just limited to the application of the ritual Islamic slaughter method, but additional conditions should be taken into account for meat to become Halal [20]. According to Tieman, Halal products or services do not only matter during the point of consumption or purchase but they transcend all the activities of the supply chain from farm-to-fork [21]. In other words, Halal status should be preserved through all the supply chain and Halal product should be protected throughout a series of movement from one supply chain channel to another channel until it reaches the ultimate customer [6]. This implies to say that Halal meat consumers have become increasingly aware and concerned about the integrity of Halal-meat related products with regard to their production, their distribution and their storage along the whole supply chain network. This could be also explained by the selective purchasing decision of Halal consumers, especially Muslims, for checking the components of the foods or drinks to ensure its non-containing of prohibited and Haram items. These latters can be split into many categories and they include any products derived from or contaminated with proscribed materials, such as, carrion, blood, pig, permitted animals slaughtered incorrectly, and intoxicants [18].

The slightest presence of non-Halal elements will breach the Halal status, thus this indicates the sensitivity and the criticality of Halal meat supply chain [14]. Kadir et al. divided Halal poultry meat supply chain into three main processes: the pre-slaughtering, the slaughtering and the post-slaughtering process [22]. The same rationale could be used in describing the different issues in Halal meat supply chain.

The pre-slaughtering stage involves any activities related to the rearing and fattening of livestock. The production of Halal meat is distinguished from any other type of foods by the inclusion of Halal feeding of livestock and the monitor of livestock health at farms [23]. Any ill or contaminated livestock should be isolated from the cattle and farmers have to keep a medical record of livestock relating to illness, medical treatments, and treatment results during the growing period [23]. Besides, any medical treatments and vaccination which contain pork enzymes make livestock non-Halal and unfit for consumption. Livestock farms should be safe and not within the vicinity of pig farms or run-off from pig farms [18]. It is also worth mentioning that Halal animals may become Haram during breeding in case the animals are supplied with any product derived from Haram sources or obtained biotechnologically through genetic engineering using ingredient and components from Haram species [24] [25]. In doing so, Halal will be humane to animals (i.e., free from epidemics, illnesses, and injuries) and environmentally friendly in addition to the healthiness, wholesomeness, and cleanness of meat. As a result, how the animals are housed, reared and fattened is the primary concern to ensure that the animal welfare is respected and the first steps of supply chain foundation are compliant to Halal guidelines.

Slaughtering is a very critical stage in Halal meat supply chain. The animals must be slaughtered according to the Sharia principles. They consist of reciting a special prayer "In the name of Allah, Allah is the Greatest" by a practicing Muslim, cutting all four tubes or vessels [18] by a sharp and clean knife which must be invisible to each slaughtered livestock, and a full bleeding of blood after slaughtering [4]. However, the use of stunning methods are very common in most non-Muslim countries (e.g., Western European countries) where there is a non-ritual slaughter that might kill the animal before slaughter and consequently render this meat prohibited or Haram [26]. Although mechanical or machine slaughter of birds is gaining acceptance among Muslims, it is undeniable that some Muslims prefer to purchase their Halal meat directly from the slaughterhouse or buy the animals from the farmer to slaughter themselves at home (which is an illegal practice in most European countries) or at the farm. This is evident in the Netherlands where 10 to 13 % of the Halal meat is bought directly from the farmer and slaughtered on the farm or at home [20]. The same practice is common in France and Belgium [20]. Many opinions in regard with stunning are still controversial. Many opponents of pre-slaughter stunning have often cited the possible death of the animal following stunning and before exsanguination which makes the pre-slaughter stunning transgress the Islamic dietary law [27]. Others attribute the retention of more blood in the carcass of animals to pre-slaughtering stunning. Nevertheless, several studies have demonstrated that there is no difference in terms of the total blood lost at exsanguination between animals slaughtered either with or without pre-slaughter stunning [28] [29]. These misconceptions yield to a lack of consensus within the Muslim community and pave the way to fraudulent activities in the Halal meat supply chain. Overall, there are different interpretations with regard to the slaughtering process in terms of the acceptance of machine slaughtering and stunning [30] but it is beyond the scope of this paper to discuss in details the Sharia requirements for stunning.

The post-slaughtering stages include the process of packaging, labeling and transporting the livestock [22]. All aforementioned activities need to comply with the Islamic Sharia [31]. Given that logistics is defined as all activities which facilitate the movement of the right product at the right time, at the right quantity, with the right descriptions and in good conditions [32], Halal status should be ensured during any activities and must adhere to the several Sharia principles.

The transportation process is a very important step to ensure the integrity of Halal logistics. It must be guaranteed that there is no cross-contamination of Halal meat and meat products with non-Halal substances such as pork carcasses. The risk of cross-contamination may happen if there is no proper segregation between Halal and non-Halal products and when the same container is jointly used for both [33]. Research has repeatedly underpinned the need for physical segregation of Halal

products to prevent any intentionally or unintentionally direct contact with elements that can taint Halal status [34] [35]. Thus, this could lead customers to doubt if the product is really Halal, which will result in rejection of the Halal product. This could be also accentuated by the perception of Muslim consumers to the high risk of contamination especially for refrigerated and bulk products (e.g., meat) [34]. In a similar vein, since the avoidance of doubt is an important concern for Muslims in both Muslim and non-Muslim countries [11], Manufacturers and suppliers can mitigate this risk by the dedication of an entire transportation system for this type of logistics activity [33]. In doing so, they will reduce the risk of cross-contamination and ease the delivery process to their customers [33].

However, the preferred level of Halal food segregation is different between Muslim and non-Muslim countries. According to Tieman et al., the Halal flaws are less frequent in Muslim countries than the non-Muslim countries, where an extensive segregation to the last mile is needed [34]. Plus, Halal logistics is more complex and costly in these countries due to the smaller volumes of Halal products destined to Muslim minorities. In view of that, [34] shows that to satisfy 80 percent of the Muslims in the Netherlands, the non-mixing of Halal and non-Halal meat in the same fridge and on the same load carrier or pallet are required for the supermarket, transport, and storage. With regard to export and import operations via sea or airport, the segregation of Halal and non-Halal meat in the same carton box is required to satisfy the already mentioned percentage.

Knowing that almost all food producers outsource their transportation activities to the third party logistics (3PL) service providers due to the low-cost operations it brings and the need to satisfy customers of Halal food products in every part of the world, it is extremely difficult and challenging to assure no breakage of Halal procedures during the transportation of the products [22]. Most logistics service providers are reluctant to provide a dedicated transport fleet for Halal meat because the volume of Halal meat for the domestic market is small [16]. As a result, they do combine the shipment of Halal meat with non-Halal meat to achieve economies of scale. Although a combined shipment of Halal and non-Halal meat can be acceptable when there is some effective separation (e.g., the use of a curtain or special cage), the cross-contamination can be minimized but not completely avoided.

Furthermore, Halal logistics is still in infant stage even in Malaysia which aspires to become the Halal hub in the future [32] and there is no a successful model of a logistics company practicing total Halal logistics [5]. This refers to the lack of the knowledge and experts in conducting conveniently Halal logistics. Plus, the efforts of educating and training logistics service providers about Halal logistics are still limited and insufficient [5]. Similarly, some logistics companies consider the training provided by Halal authorities insufficient, not progressive or irrelevant to their business [5]. As interestingly aside from this, there is a lack of collaborative efforts among the logistics service providers. The integration between conventional logistics and Halal logistics service providers is weak and there is a possible inclusion of Haram or doubtful substances in the products in the supply chain and thus the information will not be disclosed or shared [5]. This could be also explained by the inconsistent practice of Halal logistics by the partners of the supply chain and hence any party which is not practicing Halal approach will temper the Halal integrity [5].

Food packaging (ranging from primary, secondary and tertiary packaging) plays a vital role in ensuring the protection, storage and hygienic of a product and it plays a key role in reducing the potential contamination [18]. In addition to the functional benefits of packaging, Halal meat and meat products must be appropriately packaged with packaging materials that are in accordance with Sharia law [36]. Likewise, the origin of the packaging materials still matters. The production of plastic packaging which uses animal-origin gelatins and the use of oil as a lubricant to assist the production of metal cans could void Halal status if they are derived from not properly slaughtered animals or from animals whose Halal status is questionable [32].

Food supply chains are highly exposed to the risk of economically motivated adulteration (EMA). It is defined as the fraudulent, intentional substitution or addition of a substance in a product for the purpose of increasing the apparent value of the product or reducing the cost of its production, i.e., for economic gains [37]. In view of this, tampering, misrepresentation of food ingredients or packaging, and the false or misleading statements made about the product are not a new phenomenon for meat. Indeed, the intentional mislabeling of non-Halal products as Halal in order to mislead consumers into buying such products is considered as an offense [27]. This is consistent with Mueller who compared the profits from food supply chain fraud to cocaine trafficking, with fewer risks [38]. Besides, the adulteration of pork is not uncommon in Halal meat and meat products and porcine derivatives are the most frequent non-Halal materials fraudulently used by meat processors [3]. This is because they are cheap and readily available [39]. Also, the adulteration and mislabeling of Halal meat is not restricted to non-Muslim countries (e.g., the European Union countries). Nowadays, there are some Muslim countries that are concerned with meat adulteration and Halal certification since meat is imported from various non-Muslim countries such as USA, Europe, and China, even so, certifications are not necessary when meat is sold fresh at easily recognizable joints [3]. In this context, [40] conducted a test of 143 processed Halal meat (beef and poultry) products in Malaysia for the presence of non-Halal proteins from pigs, rats, cats, donkeys, dogs and other undeclared permitted species. They found 78% of mislabeled products and buffalo DNA was detected in 40 out of 58 products labeled as beef, while 33 out of the 58 products contained undeclared chicken.

Even though Halal logo or certificates contribute to reassure Muslims about the compliance of food products they consume to Islamic dietary guidelines, consumers are still skeptical about the authenticity of Halal logo or certificates [4]. Several cases decoy the trust of Halal consumers in many countries and among them Malaysia. Many food traders have been caught displaying fake certificates or self-made Halal logo which tarnished the reputation of Halal industry [4]. Besides, the fake and unauthorized certifications are explained by the inefficient, time consuming and costly certification processes [41] [42] and the corruption among certification must be smooth and not too strict. [45] suggested that Halal logo or certificates must be authorized and issued by a credible Islamic organization in order to prevent fraudulent, fake and misleading logo or certificates.

It is acknowledged in the literature that among the most important issues in relation with Halal logistics are the lack of Halal certification authority and synergy by Halal authority agency in implementing the enforcement to logistics players [33]. In fact, there are scarce standardized Halal guidelines and an absence of a worldwide Halal certification such as a universal Halal logo. This yield to many challenges in maintaining Halal integrity throughout the supply chain [5]. Besides, there are prevalent issues regarding Halal regulation and enforcement in non-Muslim countries. Halal governance is under the supervision and control of several private entities [35] [46]. This has resulted in the existence of multiple bodies offering Halal certification services [22]. That is to say, any organization is able to provide or claim to provide Halal certification and inspection services for the manufacturers that want to tap the Halal market. That's why there is a growing skepticism among Halal consumers and manufacturers about the trustworthiness and credibility of these certification bodies [22].

# **3.** Halal Meat Supply Chain and Halal Critical Control Points (HCPs)

#### 3.1. Halal Meat Supply Chain and Process Flow

Generally, Halal food supply chain refers to the process of supplying Halal food from farm-to-fork while preserving its Halalness. According to [47], it is defined as the process of managing the procurement, movement, storage and handling of materials, parts, livestock, semi-finished inventory food and non-food and related information as well as documentation flows across the supply chain in accordance with the general principles of Sharia law. With regard to this context, the typical Halal meat supply chain consists mainly of the following various actors as shown in figure 1: farmers or breeders, abattoir, meat processors, wholesalers, distributors, retailers, and final consumers.

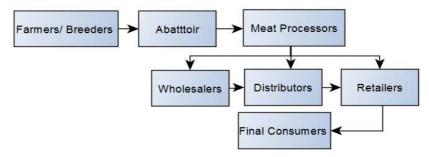


Figure 1. The Typical Halal Meat Supply Chain

The farmers or breeders start the upstream flow in the chain. In the preslaughtering stage, they play the role of livestock custodian (from young livestock to ready for slaughter). The background environment of farming should be assessed, including the distance from the vicinity of pig farms in case of a diverse livestock rearing, the welfare of the animals and the prescribed vaccinations. The livestock will be transported to the abattoir by the carriers and reasonable measures regarding the safety of animals should be ensured. In the abattoir, all slaughtering processes should be carried according to the Islamic law. As stated in figure 2, it is obvious that the real threat concerning the Halal critical control points within operations of the meat supply chain occurs at the slaughterhouse.

Five out of the ten critical control points identified in this study are found in the slaughtering stage. Besides, it is the responsibility of a Halal supervisor in the abattoir to crosscheck the health of the animals and separate the sick and injured ones. After a judgmental selection made by the Halal supervisor, the animals will be delivered for the slaughtering process. This is a key stage as differentiation between Halal and non-Halal meat occurs at this point [49]. The animal will be slaughtered one at a time. Stunning methods must be clearly administered and the time gap between stunning and slaughtering should be sufficient enough to let the animal die with bleeding in order to avoid the cross-contamination between Halal and non-Halal slaughtering. In case of a manual slaughter, which is the preferred method for cattle, the knife should be very sharp, clean and the slaughter should be performed by a Muslim and by a single swipe in order to reduce pain as much possible. Otherwise, the machines and equipment used for slaughter have to be cleaned and dry to avoid any contact with non-Halal products [50]. For the birds, mechanical slaughter is gaining acceptance among Muslims in Western European countries [20].

The invocation is a recitation of special prayer pronounced by a Muslim slaughterman while cutting the throat of an animal or while switching on the slaughtering machine. In addition, one Muslim slaughter person is positioned behind the machine to perform the slaughter if the machine misses a bird or if the cut is inadequate for proper bleeding [20].

After the complete bleeding, the animal will be skinned and its internal organs will be then removed. In this step, the meat inspector will perform the necessary inspection of the internal organs to check for any abnormalities in the cattle. The same applies to the scalding of poultry but without the need for an internal inspection. A trained slaughter person is preferred for efficiency and minimization of skin and carcass damage [47]. Immediately after that, the Halal logo will be stamped on the carcass using a special ink before it is sent to the chiller room to be cooled overnight [48].

A Halal dedicated transport logistics operator is preferred to carry out the shipment of carcasses from the abattoir to the meat processors. This practice helps to reduce the risk of cross-contamination. At the meat processors, great care should be taken during cleaning, deboning, mincing, cutting, packaging and labeling. Packaging materials should be in accordance with the Halal guidelines. Plus, the final meat products are packed and Halal logos will be displayed on the different levels of packaging and the Halal meat will be stored in good working practices while waiting for shipment to wholesalers, distributors and retailers. Eventually, retailers must preserve the Halal integrity while meat is displayed for sale and not mixing Halal with non-Halal meat products. Any inquiry from the customer to verify the origin of the Halal meat should be welcomed by the seller by granting him the right to look at the Halal meat certificate.

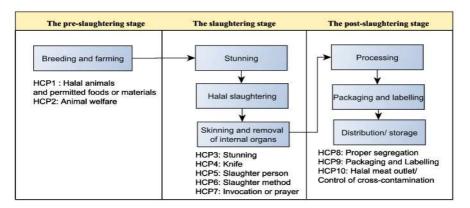


Figure 2. The Process Flow of Halal Meat and Halal Critical Control Points (HCPs) (Adapted from Riaz and Chaudry [47])

#### 3.2. The application of HACCP in Halal Meat Supply Chain

The HACCP is a systematic approach which focuses on risk management and prevention to ensure food safety [15]. This implies to say that it aims to identify, assess and control hazards during production, processing, manufacturing, preparation, and use of food to ensure its safety when consumed [48]. Apart from management tools used to assure food safety, the focus of quality assurance schemes has become more comprehensive approaches allowing the assurance and safeguarding of process standards, relating for instance to animal welfare and certified production methods such as organic or Halal [20] [49] [50] [51] [52]. In this respect, Halal Assurance System (HAS) is presented as an internal mechanism to monitor, control, improve and prevent any non-compliance in Halal production [53]. HAS is based on the concept of Total Quality Management (TQM) [54] with the inclusion of three main elements; zero limit (Halal material used in the production), zero defect (Halal product is produced) and zero risk (risks of Halal integrity breach). The principles of HAS are similar to HACCP approaches but with the added value of monitoring and control Halal status throughout the supply chain. Therefore, Halal control activities and assurance are essential to establishing a robust Halal food supply chain less vulnerable to Halal contamination [55]. Several critical points have been introduced by [56] at the slaughterhouse level to ensure the Halal status of meat. Nevertheless, an embedded Islamic dietary law into HACCP integrated system would require that the entire Halal meat chain is controlled according to HACCP principles with zero tolerance level at the implementation of corrective actions since any deviation (i.e., contamination with Haram materials) is deemed unacceptable from religious standpoint [57]. Figure 2 presents an integrated

quality assurance system with Halal critical control points. These points pertain to the whole Halal supply chain and if they are identified, controlled and monitored, they will prevent the possibility of Halal non-compliance. The Halal critical control points are identified with their control measures then followed by a table which will summarize the monitoring procedures for each hazard and the required corrective and preventive actions.

*Risks of the inclusion of non-Halal animals and unpermitted foods or materials* (*HCP1*): The animal must be within the category of Halal species allowed to be consumed in Islam. Non-Halal-animals are not permitted even if they are slaughtered in a Halal manner [45]. No genetic cross-contamination from Haram species is allowed or the use of Genetically Modified technology to derive genes from non-Halal animals. Halal animal feed is important in the Halal chain. Muslims are prohibited from consuming animals that have eaten filth [20]. Besides, carnivores and animals fed with animal protein are strictly forbidden. Ingredient in the feed mill must not contain any genetically modified organism from unpermitted animals [20].

*Risks associated with animal welfare (HCP2):* Islam stresses out the humane treatment of animals before, during and after slaughter. Animals must be in good health, not stressed or excited before slaughter. Plus, they have to be well-nourished. The knife must be invisible to station [31]. No animal should be able to witness the slaughter of another animal [20].

*Risks related to Stunning (HCP3):* Since stunning is commonly used in many non-Muslim countries to make the animal insensible and thus to inflict less pain for it. However, stunning should not render the animal dead and if the animal is not slaughtered after a certain time it will regain consciousness.

Safety risks associated with a knife and related equipment (HCP4): The knife must be clean before slaughter, flaying, and dressing. Ensure all equipment properly maintained (knife sharpness, proportionate size, cleanness of rotary knife slaughter...). Utensils, equipment, and machinery (including all food contact surfaces) must be cleaned, sanitized and untainted by contact with Haram materials [58] [57]. Chemicals used for cleaning should be screened to avoid animal fat origin [56] and brushes should be Halal certified since it can be made of pig's hair [59]

*The inclusion of non-Muslim slaughter (HCP5):* Slaughter should be performed by an adult Muslim with a sound mind [56] and without gender distinction. Jews and Christians can be involved in Slaughter since the prophet Muhammed used to eat meat prepared by Jews and Christians [11]. The meat of an animal slaughtered by an idolater, a nonbeliever, or someone who has apostatized from Islam is not acceptable [56].

Safety risks associated with slaughter method (HCP6): It is necessary that slaughter method should be done humanely and avoids the unnecessary pain of the animal. The throat must be cut with a swift blow severing the carotids, jugulars, trachea, and esophagus and without reaching the bone. The animal skinning and removal of internals before deboning should be carried properly to maintain the safety of meat and preserve its quality. Halal carcasses should be cooled in a convenient and cold storage environment.

*Risk of Invocation inaccuracy (HCP7):* A supplication must be invoked while slaughtering the animal. In doing so, the slaughterman recalls his responsibility in respecting the prescribed method of ritual slaughter and in removing any doubt as to whom the animal is dedicated [60]. Besides, it reinforces the notion of sacrificing the animal to the Almighty God. Any invocation of another name than God renders the meat Haram [20].

*Risks associated with segregation (HCP8):* In the usual case, meat processors outsource the shipping of the carcasses from the slaughterhouse to their facilities or they rely on their own transport vehicles. The refrigerated containers of the vehicles used to transport carcasses must be cleansed thoroughly. The storage conditions of the meat should be inspected by the related inspector to ensure the appropriateness of storage condition (temperature, sound segregation in case of non-Halal logistics service providers). During transportation, Halal meat must be handled properly to avoid the risk of cross-contamination.

Safety risks associated with packaging and labeling (HCP9): Site assessment as part of assured scheme (e.g., temperature controlling, disinfecting, and equipment). All processing practices should be according to good working practices, and process activities should be aligned with Halal guidelines [15]. The packages and cartons should be labeled with appropriate statements (e.g., 'keep refrigerated') to inform distributors and retailers about the necessity of keeping the product cold [61]. For meat to be packaged and labeled properly as Halal, previous Halal control points should be evaluated by a reputable supervisory organization, which acts as a trusted third party and independent certification body [20].

Safety risks associated with Retail Management (Halal meat outlets) (HCP10): Retail management and distribution are a critical issue to prevent the risk of crosscontamination. Retailers have to ensure the Halal status of meat at their retail outlets. Besides, all retail management practices should be according to working practices and should preserve Halal integrity (e.g., proper refrigeration, checking the lifetime, separation of the expired meat products, and prevention of meat intermixing, etc.).

Control	Monitoring procedures	Corrective actions
HCP1	Regulator's approval, routine reassessment, selection of persons by the reference standards and by legislative and religious requirements.	Fiqh reasoning related to GM technology; reassess site designation; document action taken
HCP2	Site documentation, inspection record, control of animal health documents, and laboratory analysis report on the disease from veterinary [62]	Separate any injured, sick or dead animal before slaughter; avoid any stressful or restraint; document actions taken
HCP3	Halal supervisor ensures proper stunning procedure. Stunning monitoring record	Review the time gap between stunning and slaughter, separate any animal suffering a fatal stunning; avoid excessive stunning voltage
HCP4	Maintenance records of the hygiene of used equipment, condition checking record	Review maintenance procedures; review workforce responsibilities (e.g. protective wears); assess the cutting surface (situation and lighting); document actions taken
HCP5	Documentation of the person responsible for the slaughter, training record, competency monitoring record and Islamic department slaughter certificate [62]	Review the workforce training since there are not enough trained Muslim slaughtermen and the market of Halal is in its infancy and demand is fleeting [56]
HCP6	Regulator's approval and assessment, visual monitoring, inspection of internal organs report	Take a regulatory control action to prevent any further improper slaughter from occurring to other animals (e.g., blood vessels raptures, muscular hemorrhage) Major pathologies in internal organs: reject the carcass and conduct a microbiological test Assurance of clean and sanitary handling and preparation ; careful working practices
HCP7	Invocation checklist	None: Invocation is a secondary condition.
HCP8	Transport monitoring, visual monitoring, Halal certification	Review procedures (selection of logistics service providers, own transportation service) ; document actions taken; review carrier Halal status
НСР9	Cleaning and disinfection checklist, microbiological control, in-house inspection of materials, proper maintenance of equipment, frequent equipment inspection	Training program for the workforce (e.g., personal hygiene, sanitary dressing and proper product handling); review the preventive maintenance program; refrigeration of perishable vacuum packaged meat; proper holding and cooling temperatures of meat; use of approved and Halal complied with packaging materials
HCP10	Site documentation	Adequate control of cross-contamination Organizing in-house transport system, order bulk volume to have less frequent delivery and full-truck-load, thus minimizing the cross-contamination risk [63] Review preventive procedures and document actions taken

Table 1. Generic HCCP management applied to the Halal Meat Supply Chain

# 4. The traceability System Based on Blockchain and Internet of Things

Since food products are scattered between the different actors of the supply chain, there are growing concerns to ensure food safety by the adoption of many internet of things technologies. Besides to being unsafe, the possibility for Halal food to be non-Halal is greater due to the travel distance whereby a lot of handling points will be included and the persistent risk of cross-contamination with non-Halal materials. To address the issues related to the stringent compliance of Halal products with Sharia law, there are a few preliminary publications in studies on the traceability of Halal meat products [31]. As a solution to that, [64] suggested a method to determine if the poultry is slaughtered according to Islamic way using an investigation of meat color. Junaini and Abdullah described a mobile-based support application for Muslims to identify Halal status [65]. Kassim et al. introduced a system to verify and recognize the information of products and thus to confirm their Halal status in real time from a real-time access to its database [66]. Bahrudin et al. proposed a comprehensive and suitable tracking and tracing technology using RFID to sustain the integrity of the Halal product and support the entire supply chain of Halal product process [47]. Iskandar et al. found that technologies such as TMS (Transportation Management System), WMS (Wharehouse Management System), EDI (Electronic Data Interchange) and GPS (Global Positioning System) are widely used among Halal logistics service providers [67]. Besides, [67] accentuated the compatibility of RFID's tracking and tracing characteristic with the Halal transportation guideline. Mohammed et al. present a framework in the development of an RFID-enabled HMSC network for enhancing traceability of Halal meat integrity throughout its entire supply chain [31]. All the previous research mentioned are the idea of using a centralized system which was latterly the only conceivable way to attain information transparency along supply chains [15]. However, there is not enough evidence about the accuracy and trustworthiness of the shared information in the traceability system and between Halal meat supply chain actors. As a result, this yields to an opaque system, information asymmetry, and many other issues. RFID- enabled traceability system is insufficient to guarantee the Halal integrity of meat. The manual retrieval and storage of information in the central database entail to many possibilities of misleading and falsification. In a similar way, it is problematic to ensure that what is termed Halal meat products are correctly compliant to Islamic dietary law and free from any misleading provenance histories. Hence, to mitigate the deficiencies of previous Halal traceability systems, we develop a conceptual framework of Halal meat BC system as illustrated in figure 3.

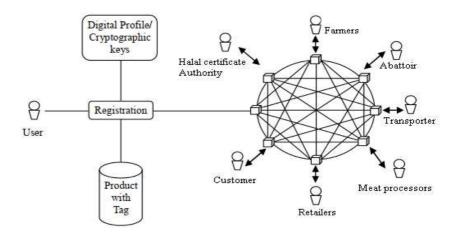


Figure 3. Conceptual framework of the Halal meat BC system (adapted from [15])

BC is a distributed ledger that becomes the foundation of a very robust system of trust, decentralization, and collaboration. The information through the system is publicly shared, replicated and permissioned. Besides, all the data in the BC is public, and any user has the access to add data in term of transaction which is identifiable data package in the system, to check and to copy this data at any time, but without the ability to change it [15]. Additionally, the BC validates the transaction without requiring intermediary assistance or the necessity of previously trusted entities [68]. Alternatively, the technology uses cryptographic or digital signature which is the trusted method to confirm the validity and finality of the transaction between the different parties [68]. Consequently, BC is a decentralized consensus system where all peers or partners come to an agreement regarding a business transaction and securely reach a state of harmony without the presence of the third party. Aside from being best-known for powering cryptocurrencies, BC technology revolutionizes and transforms the regulatory oversight of business transactions and thus it expands its seamless success to be incorporated into other industries. Similarly, this provides unprecedented opportunities for Halal industry to overcome the inherited problems (e.g., trust concerns, cross-contamination, frauds, tampering and falsifying, and fake Halal logos...) in the supply chain and logistics by the adoption of this ground-breaking and disruptive innovation.

Halal meat supply chain can be the best application scenario to show how actions in HACCP and previous RFID-enabled systems can be leveraged by the proposed BC-based traceability system. So, all Halal meat supply chain participants should register themselves in the peer to peer network and match their identities and digital profiles in back-end database which maintains an openly distributed ledger that can be accessed and inspected at any time and from any location [68]. After registration, each actor of the chain (i.e., node) will be provided with two different keys for encryption and decryption, also respectively known as a public key and a private key [69] to digitally sign each operation on the distributed ledger. The public key serves to identify the user within the system and the private key can be used for authentication when interacting in the network [70]. The different links involved in Halal meat supply chains are: Farming, Slaughtering, Meat processing, Distributing, Retailing, Certification

#### 4.1. Farming

Farmers store in the BC information about each livestock. Each animal will be attached with wearable RFID tags or sensors and will be assigned to a digital profile. Besides, the use of RFID sensor tag can automatize the data entry process related to each livestock's physical condition (e.g., heartbeats, body temperatures, breath, sweat). Plus, these devices, if build precisely and used correctly, can provide a timely diagnosis of potential animal diseases, eventually decreasing economic losses [71]. The gathered information by RFID readers is sent to a host computer management system and there is a constant flow of up-to-date and real-time information in the digital profile of each livestock in the server. In doing so, the farm monitoring will be conducted easier than using writing notes, keeping farm diary, or using simple equipment without data-sharing function [71]. Animals' food intake and nutritional requirements will be monitored in such a way that the ingredients and the blend of feeds and fodder make it very safe and free from any Haram substance. The watering points or basin can be installed with water sensors, while smart contracts can autonomously fire, hence alerting the farmers about the record of water contamination (i.e., when sensors' values are outside certain threshold) and the necessity of separating contaminated livestock from others. In addition, the farmer maintains a medical record of each livestock relating to vaccination, illnesses, and medical treatments during the fattening period. All the information of given medical treatments and vaccination should not contain any non-Halal material (e.g., pork enzymes) [31]. The Halal meat supply chain starts at the farm and the farmer generates the genesis block and adds the required information about the livestock as described in the figure 1. The block is verified by the registers in the BC system before the next block being added to the chain. Each farmer has its own identifier (ID) in the distributed ledger since there might be many farmers who are supplying meat processors. In the generic block, the farmer puts all the aforementioned information to be shared with other supply chain actors and to be verified and added to the chain.

ID: CC01			
Block 1			
Info category	Info details		
Category	Beef		
Feeding methods	Halal		
Types of diseases/symptoms	Bovine ephemeral fever		
Treatment duration	4 days		
treatment results	Healed		
Growing history/Kg	10 Kg/8 mth		
Enzyme history	None		
Last update of info	June 19, 2017		
Breeding date	01/01/2017		
Harvest date	01/09/2017		
Prev. Hash: abcdef0000a0b0bbcdcdfddfa56781ab			
New Hash: 8bc9ef5080b0affbadadfedea56481cb			

Figure 4. Typical generic block generated by the farmer (Adapted from [31])

#### 4.2. Slaughtering

After transporting livestock to the abattoir, information data of each livestock will be gathered and stored automatically into its profile by scanning their tags through an abattoir's RFID-reader mounted gate. Violations related to the employment of unaccredited slaughtermen should be addressed by abattoir supervisors. In order to comply with the Halal slaughtering process, slaughtering stations have to be monitored by abattoir operators through installed cameras [31] to enable continuous control of the activities that pose a high risk to animal welfare and Halal integrity such as the stunning area. If the animal is not slaughtered according to Sharia law, it should be separated and marked as non-Halal. At the end of slaughtering, each segmented carcass is tagged with a new RFID sensor to keep a continuous record of pH values. The information can be collected by an RFID handheld reader and subsequently entered into a new block which is visible, transparent, and verified by all the participants in the BC system. This approach will enable Halal meat supply chains to attain chain-wide transparency. Many answers to questions regarding the slaughter stage will be provided and accessible to all actors through the BC such as the stunning method, the abattoir location, the responsible staff for slaughtering and meat inspectors, and the compliance to Halal requirements. Thus, the shared ledger can be viewed by meat processors, retailers, supermarkets, carriers and Halal certification authorities.

#### 4.3. Meat processing

After receiving carcasses, meat processors start by cleaning, deboning, and cutting the meat into slices to be repacked in smaller sizes and attached with new 2D barcodes. The handheld computer's barcode scanner will record the relevant information of the packed meat product. This information includes the processing environment, processing equipment, temperature control, basic information for processing enterprise and the involved personals [15]. The use of 2D barcode is explained by their ability to store a large amount of information as machine-readable and their operability as portable databases which can be scanned and decoded by camera-equipped mobile devices [72]. Besides, the transfer of RFID data to 2D labels resulted in very high accuracy in tracing the meat through the supply chain [75]. Meat processors may input the production data about their Halal meat products in the BC allowing potential consumers to access Halal meat's data by entering barcodes into the BC or using a mobile scanner. Not only that, Halal BC will faster the flow of information and thus of materials between the meat processors and their customers (e.g., retailers, supermarkets). This is attained by triggering automatic immediate actions based on the smart contract's terms such as meat products' order, release of funds, communication, informing of Halal certification authorities and authority organizations. As such, business transactions will be automatized in a secure and decentralized manner, reduce lead-times and solve Halal issues related to reputation damage. For instance, to counter the risks attributable to the shipment of Halal meat to customers, a smart contract can be the solution to ensure that the payment by the customers will be only released if the logistics service provider confirms the delivery of Halal meat in appropriate conditions, with proper segregation and without any cross-contamination with other non-Halal meat inside the refrigerated vehicle container [70].

#### 4.4. Cold chain distribution

In the distribution process of Halal meat products, meat processors transfer the ownership of the processed meat products to distributors and retailers through the BC. Third-party logistics service providers are responsible for ensuring that Halal meat is delivered in a dedicated transportation unit that preserves Halal integrity. This is can be done by the use of vehicles equipped with a safety system (e.g., RFID reader, temperature sensor, GPS) which controls the temperature and the humidity in the different areas in the refrigerated containers. Besides to the 3T principle (i.e., Time, Temperature and Tolerance), a zero tolerance practice is adopted to ensure that the religious requirements are stringently respected [57]. Relying on these technologies, real-time environmental data of Halal meat (e.g., temperature and humidity) can be added to their digital profiles and tags at a regular time intervals [15]. Not only that, sensors (e.g., GPS and temperature sensors) and smart contracts

can automatize the process, update the meat products' digital profiles whenever anomalies are detected during the distribution phase, and send notification alert to the driver about the security threshold. In addition, the GPS system helps to position refrigerated trucks from remote distribution centers and help to optimize short delivery time in a way to guarantee the freshness of Halal meat products. The delivery and storage condition in the refrigerated container should be inspected and verified by the related staff in the BC to ensure that there is not improper physical segregation (i.e., in case there is not a Halal dedicated logistics service provider) which could, therefore, increase the trust in the Halalness of meat and avoid crosscontamination with Haram products. Eventually, BC can enhance the long-term relationship between logistics service providers and their customers in term of collaboration, goal congruence, information sharing, digitalization of paper flows, consolidation of Halal cargo, new value-added logistics and services, and more efficient settlement. Likewise, BC can also leverage the use of innovative logistics concept such as Halal cargo boxes.

#### 4.5. Retailing

Retailers and supermarkets receive Halal meat products and store them in the BC. Customers can use their phone cameras or the available RFID reader to obtain the basic information about Halal meat products when they are shopping by scanning their barcodes. Thus, consumers will be able to transparently verify the whole history and provenance of Halal meat products and support their purchase decision-making. Besides, thanks to BC technology, retailers can have the access to a unique digital product memory for not only the Halal meat products but also for the animal from which meat is derived, the status of the used equipment, and all Halal inspections used throughout the entire supply chain. Similarly, BC provides a robust traceability system resistant to falsification and immutable. It ensures the Halal integrity of the meat and meets consumers' expectations and trust concerns by enabling each supply chain actors to retrieve easily the whole history of Halal meat products. Therefore, all Halal meat supply chain actors will be involved in a decentralized system which eradicates trust problems (e.g., fake Halal logos, Halal integrity breach, tampering, and misleading product's information) and promotes more openness, transparency, reliability and security [70].

#### 4.6. Halal Certification Bodies

Throughout the different phases of the Halal meat supply chain, Halal certification bodies will benefit from BC through the easy editing of Halal meat supply chain and faster service to involved participants. Problems with regard to long Halal certification procedures, claims of Halal integrity breach and food safety hazards will be easily and instantly verified in the BC in a transparent way and with a rapid trace-back capability. Overall, BC can address the scarcity of a globally recognized Halal certification bodies by consolidating their efforts with other supply chain actors to respond to Halal issues and crisis.

#### 5. Conclusion

In this paper, we addressed the different issues and problems pertaining to Halal meat supply chain management and logistics. To respond to the increased interest for Halal products and the growing concerns of Muslim consumers toward the integrity of Halal food in general and meat in particular, we proposed a new decentralized traceability system based on BC and internet of things (i.e., RFID). Besides, an application scenario was presented to integrate food safety and Halal under HACCP. To promote the efficiency and the success of the HACCP-based Halal quality-assurance system for application to slaughtering, meat processing and distribution, an environment that reduces the risk of contamination and Halal integrity breach is necessary to protect Halal meat from various hazards that render food not only unsafe but non-Halal and unfit for consumption by Muslims. Therefore, IoT technologies (e.g., RFID and sensors) are considered as key enablers for the traceability and the monitoring of Halal meat through all its intermediate phases and from farm-to-table till it reaches the final consumers. To address the problems of centralized systems in previous studies about Halal meat traceability, we propose a BC based-system which will provide more visibility over real-time information to all Halal meat supply chain partners on both the safety and the Halal integrity of meat products. This new traceability system overcomes the risks posed by the centralized information systems and brings more openness, transparency, reliability, and security. BC may have the potential to revolutionize Halal industry and promote the success of Halal meat supply chain by leveraging the collaboration between all parties of the value chain (i.e., farmers, meat processors, distributors and retailers, logistics service providers, and Halal certification bodies). Plus, it helps to ensure the higher compliance to Halal guidelines in farming, slaughtering, processing, distribution and the downstream requirements for more Halal products' authenticity and food safety. Future research may consider the development and discussion of BC-based traceability systems for other Halal markets, namely cosmetics, Halal tourism, and pharmaceuticals.

## Aknowledgement

I would like to acknowledge and recognize the generous financial support of the project EFOP- 3.6.1-16-2016-00017- Internationalisation, creation of teaching, research and student supplies, development of knowledge and technology transfer as the means of intelligent specialization at Széchenyi István University.

# References

- [1] D. Sack, Whitebread Protestants: Food and Religion in American Culture, St. Martin's Press, New Yourk, 2000.
- [2] S.Z.S. Marzuki, C.M. Hall, P.W. Ballantine, Sustaining halal certification at restaurants in Malaysia, in: C.M. Hall, G. Stefan (Eds.), Sustaiable Culinary Systems, Local Foods, Innovation, Tourism and Hospital, 1<sup>st</sup> Edition, Routledge Taylor & Francis Group, London, 2013, pp. 256–274.
- [3] K. Nakyinsige, Y.B.C. Man, a Q. Sazili, I. Zulkifli, a B. Fatimah, Halal Meat : A Niche Product in the Food Market, 2012 2nd Int. Conf. Econ. Trade Dev. IPEDR Vol. 36, IACSIT Press. Singapore Halal. 36 (2012) pp. 167–173.
- [4] M.H. Zulfakar, M.M. Anuar, M.S.A. Talib, Conceptual Framework on Halal Food Supply Chain Integrity Enhancement, Procedia - Soc. Behav. Sci. 121 (2014) pp. 58–67. doi:10.1016/j.sbspro.2014.01.1108
- [5] M. Syazwan, A. Talib, L. Rubin, V. Khor, Qualitative Research on Critical Issues In Halal Logistics, J. Emerg. Econ. Islam. Res. 1 (2013) pp. 1–20.
- [6] M.H. Zulfakar, C. Chan, F. Jie, Institutional forces on Australian halal meat supply chain (AHMSC) operations, J. Islam. Mark. 9 (2018) pp. 80–98. doi:<u>10.1108/JIMA-01-2016-0005</u>
- [7] S. Kabir, Growing Halal Meat Demand: Does Australia Miss Out A Potential Trade Opportunity? Econ. Pap. A J. Appl. Econ. Policy. 34 (2015) pp. 60–75. doi:<u>10.1111/1759-3441.12101</u>
- [8] J.A.J. Wilson, The halal phenomenon: An extension or a new paradigm? Soc. Bus. 4 (2014) pp. 255–271. doi: <u>10.1362/204440814X14103454934294</u>
- [9] M.S. Ab Talib, S.S. Md. Sawari, A.B. Abdul Hamid, T. Ai Chin, Emerging Halal food market: an Institutional Theory of Halal certificate implementation, Manag. Res. Rev. 39 (2016) pp. 987–997. doi:<u>10.1108/MRR-06-2015-0147</u>

- [10] S.Z.S. Marzuki, C.M. Hall, P.W. Ballantine, Restaurant Manager and Halal Certification in Malaysia, J. Foodserv. Bus. Res. 15 (2012) pp. 195–214. doi:<u>10.1080/15378020.2012.677654</u>
- [11] M.M. Hussaini, Islamic Dietary Concepts & Practices, 1st Edition, Tyndale House Publishers, Bedford Park, 1993.
- [12] N. Mohamad, C. Backhouse, A Framework for the Development of Halal Food Products in Proceedings of the 2014 International Conference on Industrial Engineering and Operations Management Bali, Indonesia (2014) pp. 693–702.
- [13] W.M. Wan-Hassana, K.W. Awangb, Halal food in New Zealand restaurants: An exploratory study, Int. J. Econ. Manag. 3 (2009) pp. 385–402. doi:<u>10.1300/J047v15n01</u>
- [14] M.S. Ab Talib, A.B. Abdul Hamid, M.H. Zulfakar, Halal supply chain critical success factors: a literature review, J. Islam. Mark. 6 (2015) pp. 44–71. doi:<u>10.1108/JIMA-07-2013-0049</u>
- [15] Feng Tian, A supply chain traceability system for food safety based on HACCP, blockchain & amp; Internet of things, 2017 Int. Conf. Serv. Syst. Serv. Manag. (2017) pp. 1–6. doi:<u>10.1109/ICSSSM.2017.7996119</u>
- [16] M. Hafiz Zulfakar, C. Chan, F. Jie, Factors Influencing the Operations of the Halal Meat Supply Chain in Australia, in: K.S. Pawar, M. Nkoma (Eds.), Proceedings of the19th ISL International Symposium on Logistics, University of Nottingham, Nottingham, 2014, pp. 1–8.
- [17] A. Mukhtar, M. Mohsin Butt, Intention to choose *Halal* products: the role of religiosity, J. Islam. Mark. 3 (2012) pp. 108–120. doi:<u>10.1108/17590831211232519</u>
- [18] J.M. Soon, M. Chandia, J. Mac Regenstein, Halal integrity in the food supply chain, Br. Food J. 119 (2017) pp. 39–51. doi:<u>10.1108/BFJ-04-2016-0150</u>
- [19] B.A. Alserhan, On Islamic branding: brands as good deeds, J. Islam. Mark. 1 (2010) pp. 101–106. doi:<u>10.1108/17590831011055842</u>

- [20] K. Bonne, W. Verbeke, Religious values informing halal meat production and the control and delivery of halal credence quality, Agric. Human Values. 25 (2008) pp. 35–47. doi:10.1007/s10460-007-9076-y
- [21] M. Tieman, The application of *Halal* in supply chain management: in-depth interviews, J. Islam. Mark. 2 (2011) pp. 186–195. doi:<u>10.1108/17590831111139893</u>
- [22] M.H.A. Kadir, R.Z.R.M. Rasi, S.S. Omar, Z.I.A. Manap, Halal Supply Chain Management Streamlined Practices: Issues and Challenges, IOP Conf. Ser. Mater. Sci. Eng. 160 (1) (2016) pp. 1-8. doi:<u>10.1088/1757-899X/160/1/012070</u>
- [23] A. Mohammed, Q. Wang, Integrity of an RFID-enabled HMSC Network, in: N. Walker (Ed.), Proceedings of the The Third International Conference on Digital Enterprise and Information Systems, Shenzhen, China, 2015, pp. 79– 86.
- [24] Halal food- production, preparation, handling and storage- General guidelines (first revision), Standards Malaysia, ms.1500.2004 (2004)
- [25] Halal food- production, preparation, handling and storage- General guidelines (second revision), Standards Malaysia, ms.1500.2009 (2009)
- [26] M.H. Benkheira, La nourriture carne'e comme frontie're rituelle. Les boucheries musulmanes en France, Arch. Sci. Soc. Relig. 92 (1995) pp. 67– 88.
- [27] A. Fuseini, S.B. Wotton, T.G. Knowles, P.J. Hadley, Halal Meat Fraud and Safety Issues in the UK: a Review in the Context of the European Union, Food Ethics. 1 (2) (2017) pp. 127–142. doi:<u>10.1007/s41055-017-0009-1</u>
- [28] R. Khalid, T.G. Knowles, S.B. Wotton, A comparison of blood loss during the Halal slaughter of lambs following Traditional Religious Slaughter without stunning, Electric Head-Only Stunning and Post-Cut Electric Head-Only Stunning, Meat Sci. 110 (2015) pp. 15–23. doi:<u>10.1016/j.meatsci.2015.06.008</u>

- [29] M.H. Anil, T. Yesildere, H. Aksu, E. Matur, J.L. McKinstry, H.R. Weaver, O. Erdogan, S. Hughes, C. Mason, Comparison of Halal slaughter with captive bolt stunning and neck cutting in cattle: Exsanguination and quality parameters, Anim. Welf. 15 (2006) pp. 325–330.
- [30] M. Tieman, Control of Halal Food Chains, Islam and Civilisational Renewal, 3 (3) (2012) pp. 538-542.
- [31] A. Mohammed, Q. Wang, X. Li, A study in integrity of an RFID-monitoring HMSC, Int. J. Food Prop. 20 (2017) pp. 1145–1158. doi:10.1080/10942912.2016.1203933
- [32] M. Syazwan Ab Talib, A. Bakar Abdul Hamid, Halal logistics in Malaysia: a SWOT analysis, J. Islam. Mark. 5 (2014) pp. 322–343. doi:<u>10.1108/JIMA-03-2013-0018</u>
- [33] N. Wazirah, R. Shah, A. Muhammad, S. Mohamad, Halal Transportation Providers for Supply Chain Management in Halal Industry : a Review Pengenalan : Penyedia Pengangkutan Halal Bagi, 1 (2016) pp. 1–12.
- [34] A. Tieman, M & Ghazali, Halal control activities and assurance activities in halal food logistics, in: Int. Halal Conf. 2012, Kuala Lumpur, Malaysia, 2012.
- [35] A.-H. Lodhi, Understanding halal food supply chain, HFRC UK Ltd, London, 2009.
- [36] K. Nakyinsige, Y.B.C. Man, A.Q. Sazili, Halal authenticity issues in meat and meat products, Meat Sci. 91 (2012) pp. 207–214. doi:<u>10.1016/j.meatsci.2012.02.015</u>
- [37] J. Spink, D.C. Moyer, H. Park, J.A. Heinonen, Defining the types of counterfeiters, counterfeiting, and offender organizations, Crime Sci. 2 (2013) p. 8. doi:10.1186/2193-7680-2-8
- [38] T. Mueller, Letter from Italy, Slippery Business, Tha Trade in Adulterated Olive Oil, The New Yorker, August 13 (2007) pp. 38-45.
- [39] A.A. Aida, Y.B.C. Man, C.M.V.L. Wong, A.R. Raha, R. Son, Analysis of raw meats and fats of pigs using polymerase chain reaction for Halal authentication, Meat Sci. 69 (2005) pp. 47–52. doi:<u>10.1016/j.meatsci.2004.06.020</u>

- [40] L.O. Chuah, X. Bin He, M.E. Effarizah, Z.A. Syahariza, A.K. Shamila-Syuhada, G. Rusul, Mislabelling of beef and poultry products sold in Malaysia, Food Control. 62 (2016) pp. 157–164. doi:<u>10.1016/j.foodcont.2015.10.030</u>
- [41] M. Abdul, H. Ismail, H. Hashim, J. Johari, SMEs and Halal certification \*, China-USA Bus. Rev. 8 (2009) pp. 22–30.
- [42] N. Noordin, M. Hashim, Z. Samicho, Value Chain of Halal Certification System: A Case of the Malaysia Halal Industry Nurulhuda Noordin , Eur. Mediterr. Conf. Inf. Syst. (2009) pp. 1–14. doi:<u>10.1108/JIMA-03-2014-0027</u>
- [43] N. Saleem, Many American meat exporters obtain halal certificate fraudulently, Gulf News. (2008).
- [44] A. Evans, At The Cross-Roads, Halal Journal, 10 (2007) pp. 14–15.
- [45] M.N. Riaz, M.M. Chaudry, The value of Halal food production, Inf. Int. News Fats, Oils Relat. Mater. 15 (2004) pp. 698–700.
- [46] S. Shafie, U. Malaya, K. Lumpur, Halal Certification : an international marketing issues and challenges, Proceeding Int. IFSAM VIIIth World Congr. (2006) pp. 1–11.
- [47] S.S.M. Bahrudin, M.I. Illyas, M.I. Desa, Tracking and tracing technology for halal product integrity over the supply chain, Proc. 2011 Int. Conf. Electr. Eng. Informatics, ICEEI 2011 (2011). doi:<u>10.1109/ICEEI.2011.6021678</u>
- [48] C. Ratanamaneichat, S. Rakkarn, Quality Assurance Development of Halal Food Products for Export to Indonesia, Procedia - Soc. Behav. Sci. 88 (2013) pp. 134–141. doi:10.1016/j.sbspro.2013.08.488
- [49] J.D. Wood, J.S. Holder, D.C. Main, Quality Assurance schemes., Meat Sci. 49 (1998) S191-203. doi:10.1016/S0309-1740(98)90048-1

- [50] A. Fearne, S. Hornibrook, S. Dedman, The management of perceived risk in the food supply chain: A comparative study of retailer-led beef quality assurance schemes in Germany and Italy, Int. Food Agribus. Manag. Rev. 4 (2001) pp. 19–36. doi:<u>10.1016/S1096-7508(01)00068-4</u>
- [51] A. Juska, L. Gouveia, J. Gabriel, K.P. Stanley, Manufacturing bacteriological contamination outbreaks in industrialized meat production systems: The case of E. coli O157:H7, Agric. Human Values. 20 (2003) pp. 3–19. doi:<u>10.1023/A:1022416727626</u>
- [52] T.A. Ten Eyck, D. Thede, G. Bode, L. Bourquin, Is HACCP nothing? A disjoint constitution between inspectors, processors, and consumers and the cider industry in Michigan, Agric. Human Values. 23 (2006) pp. 205–214. doi:<u>10.1007/s10460-005-6107-4</u>
- [53] A.A. Rahman, M. Abdul, Establishment of Traceability practices through Halal Assurance System (HAS) Implementation, 7 (2017) pp. 130–139. doi:<u>10.6007/IJARBSS/v7-i6/2951</u>
- [54] S. Cerani, N. Bo, Possibilities and significance of the implementation of halal assurance system (HAS) in exixting quality system in food industry, Biotechnol. Anim. Husb. 25 (2009) pp. 261–266. doi:<u>10.2298/BAH0904261C</u>
- [55] J. V. Vlajic, J.G.A.J. Van Der Vorst, R. Haijema, A framework for designing robust food supply chains, Int. J. Prod. Econ. 137 (2012) pp. 176–189. doi:<u>10.1016/j.ijpe.2011.11.026</u>
- [56] M.N. Riaz, M.M. Chaudry, Halal Food Production, 1st Edition, CRC Press, New York, 2003.
- [57] Kohilavani, W. Zzaman, W.W.N. Abdullah, A.Y. Tajul, Embedding Islamic dietary law into an HACCP approach for application to the poultry slaughtering and processing industry, Int. Food Res. J. 22 (2015) pp. 2684– 2690.
- [58] A.N. Lau, M.H. Jamaludin, J. Mei Soon, Quality assurance and halal control points for the food industry, Nutr. Food Sci. 46 (2016) pp. 557–570. doi:<u>10.1108/NFS-03-2016-0026</u>

- [59] J. Fischer, Manufacturing halal in Malaysia, Contemp. Islam. 10 (2016) pp. 35–52.
   doi:<u>10.1007/s11562-015-0323-5</u>
- [60] A. M, Muslim attitudes to the slaughter of food animals, Anim. Regul. Stud. 3 (1981) p. 5.
- [61] R.B. Tompkin, HACCP in the meat and poultry industry, Food Control. 5 (1994) pp. 153–161.
   doi:<u>10.1016/0956-7135(94)90075-2</u>
- [62] T.A. Yang, N.A. Febrianto, W. Nadiah Wan Abdullah, A. Tajul Aris, A Decision Tree Based Approach for the Identification of Halal Critical Control Point for Slaughtering According to Islamic Dietary Law, Internet J. Food Saf. 14 (2012) pp. 48–53.
- [63] M.H. Zulfakar, Australia's Halal Meat Supply Chain (AHMSC) Operations: Supply Chain Structure, Influencing Factors and Issues, RMIT University, Melbourne, Australia, 2015.
- [64] M.A. Mohamad, S. Mansor, N. Ahmad, W.A.W. Adnan, I. Muhammad Wali, The reliability of halal product transportation using gps tracking system, J. Theor. Appl. Inf. Technol. 90 (2016) pp. 188–196.
- [65] S.N. Junaini, J. Abdullah, MyMobiHalal 2.0: Malaysian mobile halal product verification using camera phone barcode scanning and MMS, in: Proc. Int. Conf. Comput. Commun. Eng. 2008, ICCCE08 Glob. Links Hum. Dev., (2008) pp. 528–532. doi:<u>10.1109/ICCCE.2008.4580659</u>
- [66] M. Kassim, C.K.H.C.K. Yahaya, M.H.M. Zaharuddin, Z.A. Bakar, A prototype of Halal product recognition system, in: 2012 Int. Conf. Comput. Inf. Sci. ICCIS 2012 - A Conf. World Eng. Sci. Technol. Congr. ESTCON 2012 - Conf. Proc., 2012: pp. 990–994. doi:<u>10.1109/ICCISci.2012.6297170</u>
- [67] M. Iskandar, I. Tan, R.N. Razali, Z.J. Husny, The Adoption of Halal Transportations Technologies for Halal Logistics Service Providers in Malaysia, Int. J. Mech. Ind. Sciene Eng. 6 (2012) pp. 10–17.

- [68] W. Mougayar, The Business Blockchain, Wiley, New York, 2016.
- [69] M. El Maouchi, Decouples: A privacy preserving solution for traceability in supply chains, M.Sc. thesis, Delft University of Technology, 2018.
- [70] A. Rejeb, Blockchain Potential in Tilapia Supply Chain in Ghana, Acta Technica Jaurinensis 11 (2) (2018) pp. 104-118. doi: 10.14513/actatechjaur.v11.n2.462
- [71] S. Neethirajan, Recent advances in wearable sensors for animal health management, Sens. Bio-Sensing Res. 12 (2017) pp. 15–29. doi:<u>10.1016/j.sbsr.2016.11.004</u>
- [72] D. Buskirk, R. Clarke, D. Grooms, K. Kirk, Development of a Farm To Consumer Traceability Model, 2011.